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ESR IS 50 YEARS!

Electroslag remelting (ESR), creation of scientists of our institute, was developed 50 years ago within walls of its laboratories. Practically at the same time at Zaporozhie Electrometallurgical Plant «Dneprospetsstal» and Novo-Kramatorsk Machine Building Works first in the world commercial ESR furnaces were commissioned. Within short time ESR started to be widely distributed in the world, and nowadays this process together with vacuum-arc remelting is the basis of special electrometallurgy. According to various estimations, about 1.2 mln t of electroslag metal are melted annually in the world, whereby half of this volume — in China. At present ESR is a standard, well studied metallurgical process that ensures production of steels and alloys of top quality.

Nevertheless, metallurgists face now new extremely complex tasks. The main point is production of high-quality gigantic solid and hollow steel ingots of up to 300 t mass (and possibly up to 400 t), which is by one order less than mass of ingots from high-temperature alloys. It would seem that these tasks are known and were more or less successfully solved almost 30 years ago first of all for needs of power engineering. However, nowadays they got significantly more complex and they have to be solved for new generation of steels and alloys. First experience of application of so called double-circuit ESR with practically independent energy input and productivity allow assuming that such solution will be found.

In recent time quite a few other interesting and complex problems occurred. For example, it was discovered long ago that in certain cases a cast electroslag metal is not inferior to the strained one and sometimes even exceeds it by service characteristics in specific items. At present acute need exists in manufacturing of cast billets of special-purpose thick-wall vessels, including those for nuclear power engineering and petrochemistry. Hear hollow ingots of up to 4 and even 5 m diameter are meant.

One should not forget at new spire of the ESR development that possibilities of refining of a metal by slag with application of not just alternating, but also of direct current, or in case of their simultaneous passage through the slag are not completely studied, and this means that field of activity is open not just for technologists, but for the researchers as well.



Academician
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SOLIDIFICATION OF METAL IN REMELTING PROCESSES

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The two remelting processes of electroslag and vacuum arc remelting are widely used in industry for the manufacture of high quality steels, superalloys and titanium alloys. Originally employed for refining, they are now used principally as solidification controlling steps in ingot manufacture. Since the boundary conditions of the process can be readily measured, many attempts have been made to model the ingot solidification. In this work we review the models and indicate their success in application. We conclude that although the present CFD models have greatly assisted in our understanding of the basic processes, as yet they are insufficient to describe the detail required for useful application in industry.

Keywords: *electroslag remelting, vacuum arc remelting, steels, superalloys, titanium, flux, slag, nonconsumable electrode, magnetic field*

The production of materials which are free from detrimental segregation has long demanded the use of the remelting processes of vacuum arc remelting (VAR) and electroslag remelting (ESR). The two processes combined now have an annual world production approaching 0.5 mln t. This total is very small when compared with the billion tones per year of conventional steelmaking, but it nonetheless represents a commercial value of alloy processed of approximately $5 \cdot 10^9$ at the ingot stage.

In the processes the alloys are essentially contained by a solid «skull» of the metal, which is in turn held in a water-cooled copper mold, the requisite heating energy being supplied by a DC arc, or more recently by electron beams or plasma. The VAR method was initially seen to have the advantage over ESR in that it can be operated to give higher cooling rates in the ingot and hence to provide less segregation than does ESR. This feature continues to account for the on-going use of the method, particularly in alloys which are highly sensitive to segregation such as the nickel-base superalloys.

In order to understand the solidification features of the processes it is necessary to outline their respective heat balances and to discuss the boundary conditions involved, both of which have been the subject of extensive computer modeling exercises. The general operating characteristics of the processes have been described in numerous reviews, summarized by Choudhury [1]. Although the two processes have strong similarities in a general sense, the details of the thermal regime are very substantially different and should be discussed separately.

ESR process thermal characteristics. Energy to ESR is supplied by line-frequency AC power which develops the required temperature through resistance heating in the slag region of the process. The function of the slag is not only to generate heat, but also to transfer the heat energy to the electrode (for melting),

to the liquid metal leaving the electrode (for superheating) and to the ingot surface for maintaining the required temperature gradients in the solidifying ingot. The precise way in which this is carried out represents the core of the technical operation, but from the point of view of solidification we may discuss only the effect of enthalpy input to the ingot.

In early computer models of the process [2, 3], the heat input was represented by an assumed boundary condition on the top surface of the ingot, either of enthalpy input or simply temperature. This assumed condition had various forms, depending on the chosen computational formulation, but in most cases was held to be an adjustable parameter. The initial input form was generally parabolic with a centreline temperature, for example, showing approximately 150 °C superheat and a peripheral boundary temperature close to the alloy liquidus. The ingot cylindrical surfaces were represented by heat transfer coefficients, or imposed temperatures. In the former case, laboratory measurements [4] were made to validate the coefficients used and in some cases measurements were also made on industrial furnaces [5] during operation. One deficiency of these early models was that none of them allowed for the small region of liquid metal contact on the slag skin at the top periphery of the ingot which is responsible for important features such as surface quality, and in the case of roll steels the angle of inclination of the near-surface primary dendrites.

A similar approach was adopted for the water-cooled baseplate supporting the ingot in the water-cooled copper mold. By using various methods of marking the extent of the liquid pool during melting it was possible to adjust the boundary conditions to obtain a reasonable representation of the ingot thermal regime and so to explain various features of the solidification process.

One method commonly used in the case of steels was the point addition of sulphur followed by sulphur printing of the ingot axial section [6] although other techniques such as the insertion of tungsten markers or the addition of radiotracers were also used [7]. (It is interesting to note that this type of modeling in-

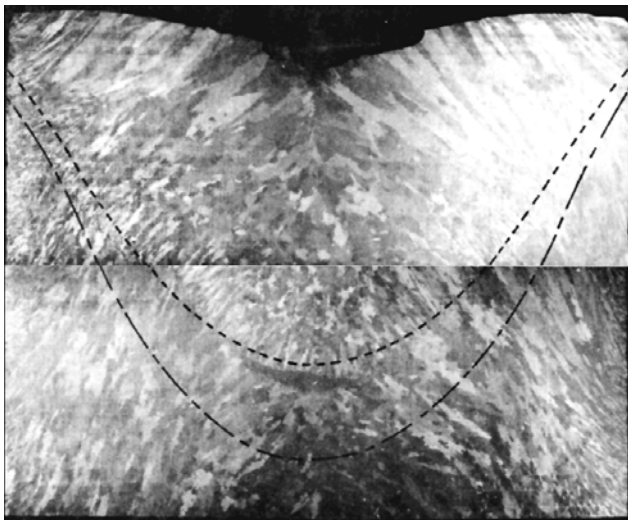


Figure 1. Computed and actual pool shape for an ESR ingot of alloy U700, 500 mm diameter (dotted line — liquidus, solid line — solidus)

vestigation on ESR preceded similar work in continuous casting.)

As a results of these modeling exercises it was clear that the modeling techniques, with a sufficient number of adjustable parameters, could adequately describe the generalities of the solidification process in both VAR and ESR (Figures 1–3).

However, it was also clear that several important details of both ingot structure and process operation could not be modeled with any level of reliability. These details included the precise shape of the liquidus isotherm, which in neither process is exactly the predicted paraboloid; the temperature gradients in the freezing zone; the behaviour of solidification through transient heating or cooling conditions; and the response of the solidification system to liquid metal movement in the fully-liquid pool. In order to develop more realistic models it was found necessary to consider the processes as a whole, including the melting and heating components as well as the freezing ingot, in which case the two processes of VAR and ESR must be considered separately.

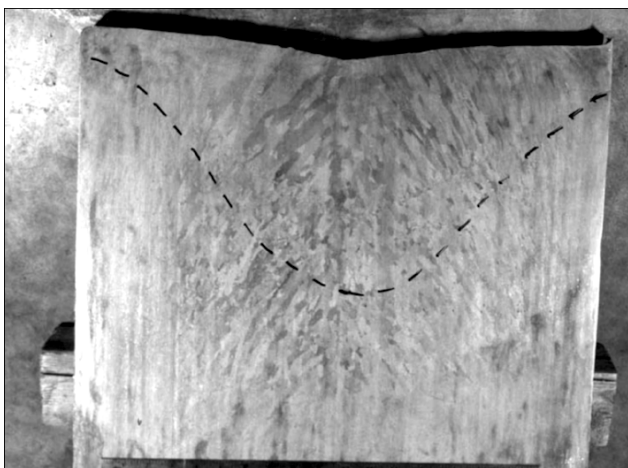


Figure 2. ESR ingot section, 500 mm diameter produced at high melting rate (dotted line — contour of molten pool)

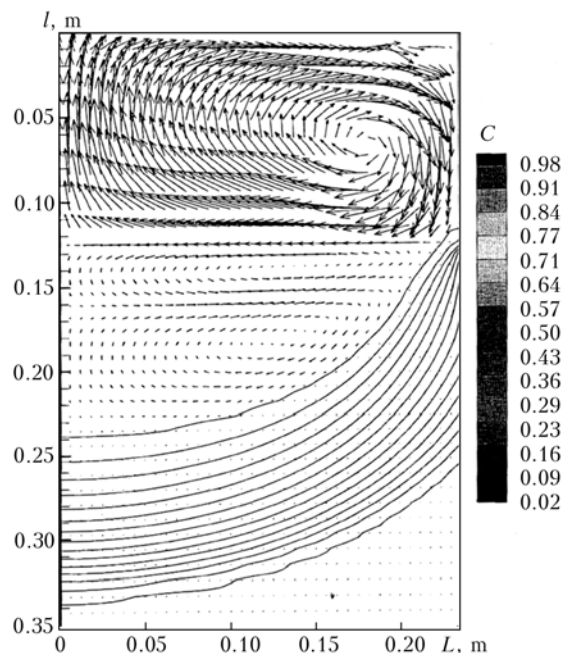


Figure 3. ESR ingot model for high melting rate: l — axial distance; L — radial distance; C — liquid fraction

The heating process in ESR was initially considered to be the uniform resistance heating of the slag, providing isotherms which could be computed on the basis of a simple axisymmetric resistance / temperature relationship. The electrode surface was assumed to have a fixed geometry, immersed in the slag with its surface at the working potential of the process; the slag axial boundary was the water-cooled copper mold insulated by a zero-conductivity solid slag skin; the ingot surface was uniformly at ground potential; and the slag itself was not in motion [2]. Although these model assumptions can obviously be challenged on the basis of actual process operational observation, the model could be adjusted to give a somewhat more realistic picture of the ingot surface temperature and hence an improvement to the ingot model results. It rapidly became clear, however, that the model assumptions were too unrealistic in view of the fact that none of the important observations of the effects for example of changing electrode diameter or of slag movement could be reproduced by the model. Nonetheless, modeling of the ingot structures was continued, on the basis of the temperature boundary conditions computed by the simple model structure described above.

The initial extensions of the «pool profile» models were directed at obtaining a more accurate computation of the liquidus profile, accounting for the non-paraboloid surfaces found in many practical instances. The effect was held to be caused by movement in the liquid pool, generated by a combination of mass flow, buoyancy and electromagnetic effects, and was accounted for by an arbitrarily-varying thermal conductivity of the liquid metal in both radial and axial directions (essentially a curve-fitting exercise). The technique permitted a force-fitting of the liquidus profile which was also assumed, when combined with



an equilibrium phase model of solidification, to give a reliable picture of the solidifying zone and hence of the anticipated metallurgical structure.

This structure was represented by the «local solidification time» (LST), i.e. the time taken for the alloy to travel between the liquidus and the solidus or the combination of the mushy-zone temperature gradient and the local solidification rate [8, 9]. The LST concept of structure description has the advantage that it permits an extrapolation from laboratory directional solidification experiments to a prediction of ingot structure without the need for expensive cutting of large ingot sections. It was shown to give reasonable predictions of structure in high-speed steels and in superalloys [10], not only in respect of dendrite spacings but also in respect of primary carbide sizes and compositions.

The output of the models based on this concept has also been used to develop models of grain structure, and predictions of the position of columnar / equiax transitions (CET) in larger ingots [11]. The latter extension of the models has been applied so far only to superalloys and not to the available data on very large ESR steel ingots [12]. In this case, the conditions are known precisely from laboratory experimentation and also from actual ingot sections and would provide a very demanding test of the model predictions. The transition conditions predicted from the models agree well with available data on the CET from directional solidification in the superalloys [13].

ESR metallurgical effects. The primary purpose of the ESR process was initially one of refining to remove, for example sulphur and non-metallic inclusions. Although the need for the former has largely disappeared except in some extreme cases of very low sulphur alloys, the latter still remains an important aspect of the operation. Investigations have shown that the ESR ingot inclusions content is largely dependent on the precipitation of secondary inclusions in the liquid pool and in the solidifying zone and hence on the pool volume and on the LST. The same investigations have also shown that the size range of inclusions found in the ingot is related to the melting rate, which amongst other aspects determines the residence time available for flotation of inclusions in the ingot pool.

The secondary purpose of the process is to control the solidification of the ingot and hence its structure. The computed LST has a direct bearing on the equilibrium structure, as indicated above, but does not directly provide information on the possibility of segregation defects such as freckles or center segregation appearing in the ingot. Since these latter defects are a very significant problem in practice, efforts have been made to refine the model structures so as to provide information which would be useful in practice to avoid the problems.

The issue of macrosegregation is complex. In principle, given the columnar-dendritic nature of the solidification any segregation should be restricted to



Figure 4. Cross-section of ESR ingot of 450 mm diameter from alloy IN718 produced at low melting rate (dashed line — actual liquidus line at melt termination)

the region between the dendrites. Experimental work on this aspect of the process, using radiotracers [7], shows that the liquid pool is fully mixed and that any ingot composition changes initiated by an abrupt composition change, for example, in electrode composition can be accounted for by using simple mixing equations. One result is the conclusion that a step change in electrode composition will result in a gradual change in the ingot composition from initial to final composition over an ingot length equivalent to three pool volumes.

The same studies also showed that there was very little exchange of liquid between the interdendritic liquid and the bulk liquid pool. This finding agrees well with the more recent results of sophisticated process models which indicate a so-called «immobilization» temperature; a temperature in the solidification zone where the fraction solid prevents any liquid exchange with the bulk above the liquidus line. This temperature is usually taken to be at approximately the point of 10–20 % solidification, i.e. a fraction solid of between 0.1 and 0.2.

Given the results of these investigations, we would expect, as is found in practice, that the composition of ESR ingots does not vary from bottom to top when a uniform composition electrode is used, provided that the solidification scheme is restricted to 100 % columnar-dendritic (Figure 4). In the case of ingots made at excessive melting rates and / or in very large diameters relative to the segregation sensitivity, where we find some degree of equiax structure, we can also find macrosegregation due to the so-called «crystal rain», where the physical movement of equiax crystals has led to macrosegregation in the same manner as in conventionally-cast ingots.

Although the conditions leading to axial macrosegregation are well understood and predictable

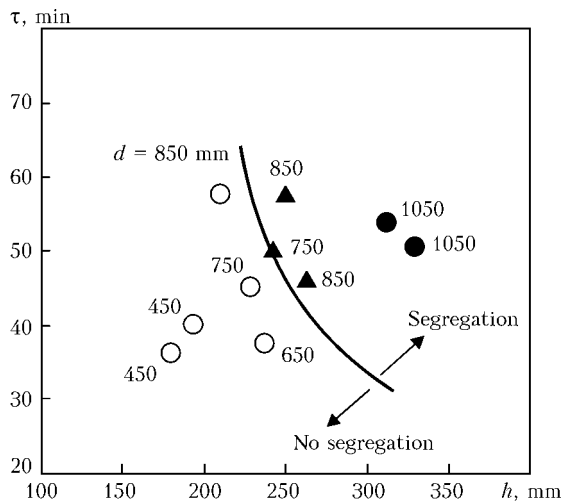


Figure 5. Boundary of centre segregation region in the ESR of alloy IN706 [16]: τ — local solidification time; h — depth of molten pool; ○ — no segregation; ▲ — slight segregation; ● — heavy segregation

from computational models, macrosegregation in the radial sense is less so. It is clear from experimental studies that there is liquid movement by flow through the solidifying dendrite matrix, and that this flow is controlled by several factors related to the solidifying region thermal regime. Interdendritic fluid flow has been studied by many authors [14, 15] and there are numerous descriptions of the governing equations.

However, it is clear that in the ESR (or VAR) situation, the primary variables are the dendrite spacings and the degree of segregation in the alloy chosen. The former is related directly to the thermal regime and is relatively insensitive to alloy composition; the latter is alloy-dependent. The centre segregation found in ESR ingots (sometimes termed «ingotism» in billet examinations) appears when the alloy being melted is segregation sensitive and is being melted at a rate which results in a critical dendrite spacing and dendrite inclination in the ingot centre regions.

These conditions are very dependent on the alloy and on the melting conditions of melting rate and ingot cross-section, but are a constant practical problem since they limit the size of ingot which can be made in a particular alloy composition.

An example of a study which demonstrates this aspect is given in [16], where the alloy in question IN706 has a moderate sensitivity to segregation principally due to niobium partitioning (Figure 5). Similar results for a wide variety of compositions resulted in industrial data which relates ingot diameters d , melting rates and alloy compositions [17]. The same principal lies behind attempts to describe the ingot solidification in various melting conditions in terms of dimensionless numbers [18], which are useful in a very general sense but do not directly lead to a knowledge of the ingot structure to be obtained.

The conditions in respect of dendrite spacing etc. which lead to centre segregation are also those which can produce the defect known as «freckle» (or «V-segregation» in conventional casting). The principal

mechanism of freckle formation, buoyancy-driven flow through the columnar-dendritic structure, has been studied intensely [19] due to the importance in maintaining billet quality free from the defect in critical alloy components.

The formulation of the limiting conditions for freckle formation is complex. Several workers [19–22] have used the technique of dimensionless number (in this case the Rayleigh number) to describe the conditions under which a freckle channel may form. The verification of this postulate has been studied in the laboratory using the technique of directional solidification and the results appear to confirm the validity of the model. The Rayleigh number can be derived for ingot ESR practice directly from the relationships between the dendrite spacing and the solidification thermal regime [23] but the application to industrial results has not yet been demonstrated, possibly due to a combination of the inaccuracy of existing thermal models, the uncertainty of the dendrite spacing / temperature gradient relationships (error bands of 30 % are common) and also due to the uncertainty inherent in our knowledge of the physical properties of liquid metals [24].

In this application a very small density gradient in the interdendritic liquid is required to drive the freckle formation (approximately 3–5 %), which is well within the error band of our knowledge of even the temperature dependence of the density of pure iron or nickel. A further complication in respect of freckle formation is that the Rayleigh number approach indicates the potential for freckle propagation and not for initiation. The latter requires some instability in the solidification to start the liquid movement, following which event the Rayleigh number concept will reliably comment on the freckle channel ability to sustain continued movement.

The initiating instability found in practice has been attributed to the production of CO bubbles [25] in steels, and also to transient liquid movement in the bulk liquid pool. In the former case, the mechanism has been verified experimentally and appears to be a significant contributor to freckle formation in ESR ingots of steels made under conditions where oxidation may occur which leads to a supersaturation of the interdendritic liquid due to carbon and oxygen segregation on freezing. This situation may be avoided by suitable reduction of the oxygen content of the ingot.

The effect of transient liquid movement in the metal bulk pool has been demonstrated for VAR, but not for ESR. In the former case, any random stirring of the liquid metal pool by stray electromagnetic fields will lead to freckle formation, given the correct Rayleigh number conditions. In ESR, freckle formation is also found when the ingot undergoes any abrupt change in thermal regime, such as a change in the current flow as described below, which leads indirectly to liquid movement. Given, however, that the concept of the immobilization temperature appears to



be valid for the description of the thermal fields in the mushy zone and that the freckle initiation site is found [19] at a fraction solid of at least 0.5, the form of the momentum transfer between the bulk liquid and the liquid at a fraction solid of 0.5 remains unknown.

Computations of the ESR thermal regime. The initial modeling concepts have been described above and it is seen that the model validity relies on two principal aspects; the definition of boundary conditions, both internal and external, and the way in which the model accounts for enthalpy input to the ingot. The form of the industrial process makes it difficult to make direct measurements of the internal conditions of temperature, but some data is available from both industrial and laboratory work [26].

The principal finding is that there is a temperature gradient in the slag phase which cannot be accounted for on a simple resistance mode, and that there is also very significant slag movement which is related to the process conditions used. A further finding is that both of these latter effects are strongly related to the degree of electrode immersion in the slag and to the relative diameters of the electrode and ingot mold. A further important variable has been found to lie in the current distribution between the electrode–ingot path and the electrode–ingot mold path. In principle, the solid slag skin which forms between the slag and the water-cooled ingot mold should be an electrical insulator and early modeling attempts to describe the slag behaviour assumed that this was the case [2]. However, subsequent industrial trials have shown that a substantial fraction of the total current moves in the latter path, even to the extent that a process variant has been developed in which 100 % of the current is forced in this path [27].

The effect of the current path on slag movement and temperature is highly significant and accounts for many of the practical observations of slag skin thickness and of ingot pool profile shapes. Early descriptions of slag movement as a function of electrode / ingot mold diameter ratio [28] show that this ratio has a strong effect on slag movement due to the relative magnitude of buoyancy and electromagnetic forces, but do not take into account the effect of electrode immersion on the current path (Figure 6).

In small laboratory furnaces, the electrode is invariably well immersed and the mold current is close to zero, which was also the case with many of the early industrial furnaces. In a modern industrial furnace, however, the operation is conducted so that the electrode is essentially «floating» on the slag surface under a condition where the melting, rippled surface has contact with the slag over approximately 70 % of its cross-sectional area. The resulting balance between buoyancy and electromagnetic forces then leads to a condition where for a large electrode / ingot mold diameter ratio the buoyancy force predominates with a strong movement outwards towards the slag periphery.

Symmetry axis

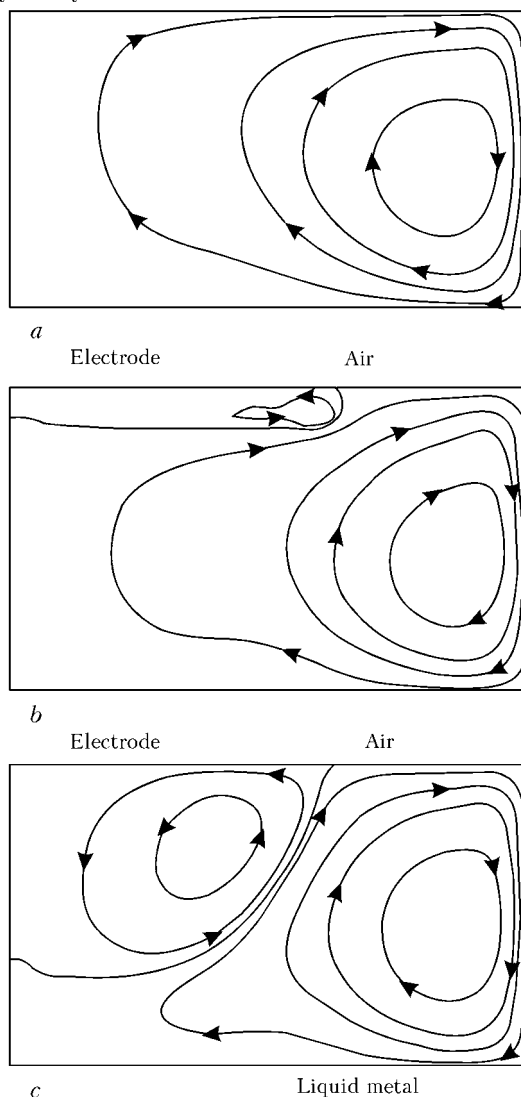


Figure 6. Effect of the electrode diameter changing on the slag flow pattern [12] at fill ration of 0.64 (a), 0.36 (b) and 0.16 (c)

The result is a high temperature at the periphery and a thin slag skin, as demonstrated by measurements of the heat transfer at this point [29] (Figure 7). This condition also provides the maximum heat transfer coefficient at this boundary for ingot contact and so modifies the ingot solidification temperature gradients in a positive sense.

The effect must be taken into account when developing a model of the ingot thermal regime, and the use of boundary heat transfer conditions taken from laboratory furnaces [30] or slag temperatures obtained from the same source [31] is probably incorrect for the present form of industrial operation. Models have recently been developed which account for all of the above effects [32] and have been verified through examination of ingot sections made on industrial furnaces. However, it is clear that progress in this direction is presently controlled by the lack of knowledge of physical properties involved (in both the slag and metallic phases) rather than by any computational problems since the software available is

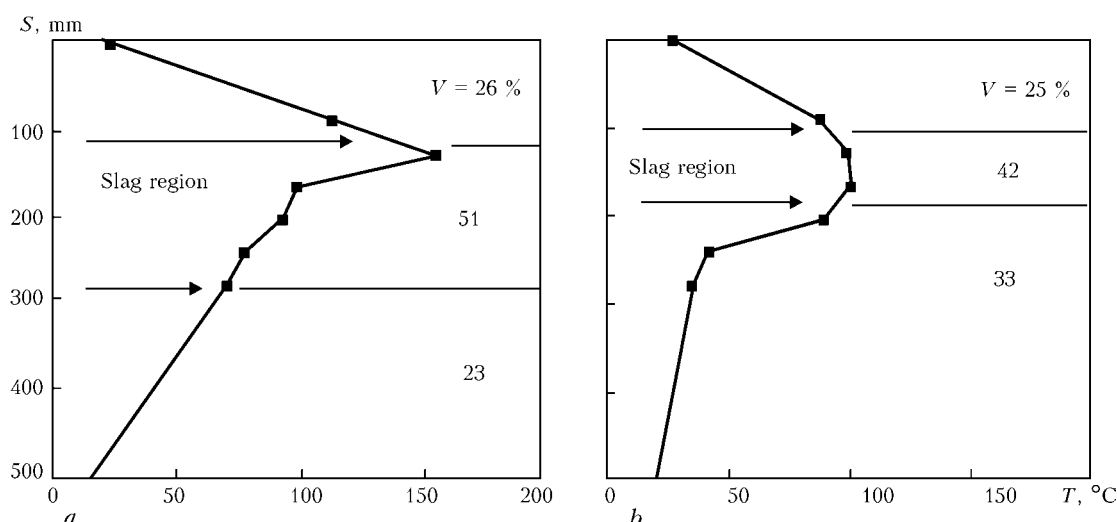


Figure 7. Heat transfer into the mold using an electrode in «floating» contact with the slag surface (a), and using an electrode well-immersed into the slag (b) [36]: S — position on mold wall; T — mold wall temperature; v — heat sharing in the melting zone

ample for the tasks involved in the process descriptions.

Descriptions of VAR process solidification. In the case of ESR, discussion can reasonably be limited to the solidification of steels and nickel-base alloys since these constitute virtually 100 % of the materials melted by this technique. VAR, on the other hand, is not only used for these alloys but also for the reactive alloys such as those of Ti, Zr, Nb, Ta and U. The major part of the work which has been devoted to investigations of solidification structure and alloy properties in VAR has been done on steels and nickel-base alloys, however, and only recently have attempts to explain features of the solidification of titanium and zirconium alloys appeared. It is clear from these latter studies that VAR as applied to those alloys is a very different process from that applied to the lower melting-point materials and the two versions should be considered separately from each other.

Concerning first the solidification of the lower melting-point alloys, the progress of computational descriptions of ingot solidification in VAR proceeded initially in the same way as for ESR, namely using boundary conditions on the ingot axial surfaces which could be reasonably measured experimentally, but also using assumed ingot top-surface temperature distributions. The results followed also a very similar pattern in that some degree of parameter fitting was required in every case in order to force a fit between the measured «pool profiles» and the predicted liquidus [33]. The ingot structures thus predicted could be analysed in terms of the relation, for example, between the dendrite spacings and the computed LST and within the accuracy of the laboratory dendrite spacing determinations, the agreement was reasonable. Nonetheless, as was the case with ESR, it became clear that most of the notable (and usually troublesome) features of the solidification could not be analysed with this method which involved too many assumed or fitted conditions.

The first of the problems concerns the representation of liquid pool movement. The flow in the

pool is driven by the same balance of buoyancy, momentum transfer from mass flow and electromagnetic interaction that is the case in ESR. In the VAR case, the electromagnetic interactions are different since the process operates with direct current, but the problem of current partitioning between the ingot and the mold paths is quite similar. In VAR, almost all of the current from the electrode tip passes directly into the mold through the top of the ingot, following a pattern which decays axially downward becoming zero at the point where contraction causes the ingot to lose contact with the mold. This point is assumed to be at the position where the hydrostatic pressure of the liquid pool can no longer deform the solid shell of the ingot into good contact with the mold, probably occurring when the ingot surface temperature falls to some 100 °C below the solidus temperature. The assumed form of this current decay has a direct influence on the fluid flow computed from the electromagnetic interactions, hence on the ingot pool temperature distribution and on the solidification temperature gradients. Some attempts have been made to measure this effect [34], but at present it remains an important undefined quantity.

The second of the problems concerns the arc heating process. The arc is a small point heat source which is intentionally moved rapidly over the ingot top surface to provide a near-uniform heat input to the ingot, which together with the fact that the electrode has a flat melting surface gives a radially-uniform enthalpy input to the ingot. The precise process conditions (arc length, vacuum integrity, electrode travel rate, electrode / mold diameter ratio) may cause this uniform arc movement to change on either a short or long timescale which both have direct effects on the solidification pattern of the ingot. The short timescale effect has characteristics which lead to the initiation of freckle defects: the long timescale can gradually permit the ingot to drift into a thermal region which leads to centre segregation. The radial arc distribution effect has been measured experimentally [34] and has



been incorporated into an ingot model in order to refine the thermal field prediction with good success.

A final problem, which is common to both ESR and VAR lies in the definition of the total energy input. Few early models attempted to run an internal check by computing the energy balance of the process and it was found that when models which incorporated the melting process component as well as the freezing component were checked against the actual energy balance, there were considerable discrepancies in the direction of computed balances which indicated far more efficient melting than that found for the actual system. In VAR, it appears that plasma discharges occur constantly along the unmelted portion of the electrode, leading to a current leakage into the mold not involving significant electrode heating. In ESR, the problem lies in the fact that most industrial power supplies do not provide a clean sinewave form of AC power, and also that some furnaces do not operate at a high power factor. As a result, the energy balance computed directly from a multiplication of voltage and current is higher than the actual power supplied to the furnace. Additionally, this factor also complicates the precise description of the slag temperature distribution in that the actual electrode surface voltage is not known directly from the applied furnace voltage measurement. The end result in both processes is that the computed solidification structure still contains unknown factors in spite of the present sophistication of the models.

Although freckles receive much attention in the scientific literature concerning VAR, in industrial practice the feature is now relatively rare since the conditions for its avoidance are well recognized. Other features provide more difficulty in quality control.

The first of these features is the defect known as «white spot» [35], which is a small (several cubic millimeters) volume of alloy having a composition which is close to that of the primary dendrite core. The defect has been carefully categorized and it appears that there are three separate types of defect. The first type originates as dendrite fragments which have fallen into the ingot pool directly from centre cavities in the electrode; the second is also fall-in, but from the «shelf» and «crown» regions of the process where splat solidification has resulted in a composition depleted in alloy elements; the third is the result of interdendritic liquid movement during solidification, a feature which might be described as an «anti-freckle». None of these features lends itself to modeling since the mechanisms are essentially the result of random process events. The defect, however, is probably the most troublesome defect in the practical production of the superalloys, and is occasionally also detrimental in alloy steels. (Since the mechanisms proposed for the various types of white spot have no counterpart in ESR, the problem is almost never experienced in that process.)

The second feature found in VAR ingots is «tree rings», which are bands of slight segregation generally

mimicking the ingot pool profile. They are common to both VAR and ESR and are the result of instabilities in the temperature regime of the ingot. These instabilities lead to an irregular progression of the dendrite tips, causing a periodic variation in the constitutional segregation existing in this region and consequently a variation in the composition of the solid frozen at the time. The causes of the instability differ between the processes, but also defy the present state of modeling in that they occur over short time intervals (and consequently short solidification distances) which would necessitate fine meshing and resulting unrealistic computing times for proper definition. It is not clear that this particular solidification effect provides any significant mechanical problems in the product, but since it is easily visible on etch examination of the product it is generally a cause for rejection.

A final feature of VAR solidification in the lower melting-point alloys is the use of helium in the space between the contacting ingot and the inner surface of the mold to enhance heat transfer. The space is a vacuum insulator, with heat transfer by radiation only, and is a significant part of the overall heat transfer resistance between the ingot centre and the water cooling. Filling the space with a helium pressure of approximately 5 kPa provides a significant decrease in the overall heat transfer resistance and so has a positive effect on the cooling rate of the central regions of the ingot. In the case of the most segregation sensitive alloys this effect translates into a 10 % increase in the maximum diameter of ingot that can be processed successfully by VAR [36]. The helium injection system is simple, but relies for its integrity on the seal formed by the ingot top periphery against the mold wall. If this seal is not valid, due to instability in the arc process, then helium will escape into the arc region and further destabilize the process leading to the defects indicated above.

It is therefore a process feature to be treated with some caution in practice. There is no parallel for the process in ESR due to the small increase in heat transfer which would be engendered by helium addition in ESR carried out under air or argon.

VAR process applied to reactive metals. The VAR process as applied to titanium- and zirconium-alloys pre-dates the application to steels and nickel-base alloys, but is also considerably less sophisticated in its present form. This situation comes about due to the relative insensitivity to segregation of most of the titanium- and zirconium-alloys which are processed industrially. Zircalloys, CPTi and Ti6 / 4 are all very insensitive to segregation and account for almost all of the reactive alloy processed by VAR. The segregation-sensitive alloys of titanium present some interesting technical challenges in VAR technology, but also represent at present only a very small fraction of the total tonnage of alloy processed.

Due to the high value of the alloy process yield is of high importance, leading to an emphasis on surface quality. The alloys have high melting points (e.g.

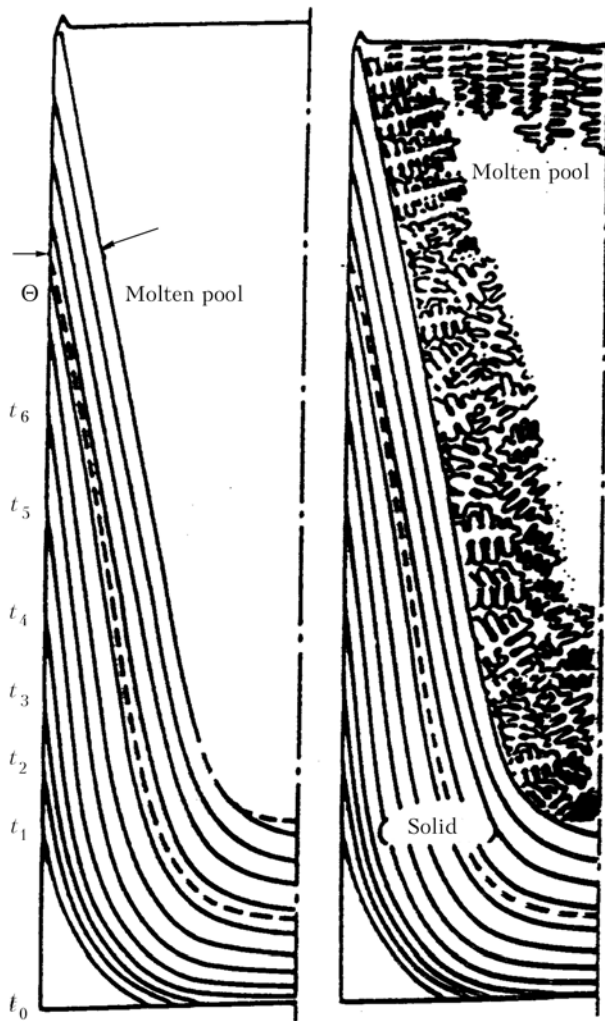


Figure 8. Titanium ingot solidification during VAR showing the CET as the ingot melting cycle transfers from steady-state to hot-top [39]

Ti — 1667 °C) and a good surface quality demands high process heat input and consequently a high melting rate leading to very large liquid pool volumes. In the melting of a large ingot of CPTi, for example, 60–70 % of the total ingot volume is still liquid at the time the process sequence enters into the hot-top cycle, in contrast with a similar steel ingot in which the equivalent volume would be less than 10 % (Figure 8). In many ways, therefore, the solidification of a CPTi ingot is more akin to that of a conventionally-cast ingot than it is to a VAR ingot of steel.

The solidification process has been modeled generally in the same way as for the steels and nickel-base alloys [37] but contains some interesting differences. The first notable difference is that the arc heat process is much simpler in representation as the arc is longer and is deliberately rotated by an external magnetic field. This feature provides a predictable uniform heat input to the ingot surface. The second notable difference is the application of a periodically reversing external magnetic field to the ingot in order to oscillate the liquid metal and provide stirring to homogenize the alloy composition. Both of these factors must be

incorporated in any model describing the solidification of the ingot.

The observed solidification pattern [38] shows a columnar structure (cellular in CPTi, but dendritic in Ti6 / 4) in the portion of the ingot which solidified whilst the steady-state melting was taking place, i.e. while the solidification temperature gradients were relatively steep. In the region of the ingot which solidified during or after the hot-top cycle, i.e. when the temperature gradients were becoming much less steep and the solidification rate was increasing, the structure is equiax. As might be anticipated, the cellular structure region of the CPTi ingot shows some macrosegregation due to solute partitioning on an essentially planar interface. Oxygen concentrates in the solid and the trace iron content concentrates in the liquid. We also observe segregation in Ti6 / 4, but in the final liquid to freeze, i.e. the ingot top central region, where aluminium has been concentrated in the top-centre due to the crystal rain effect in the equiax region. Both of these effects have been successfully modeled with the interesting finding that the CET in CPTi takes place under conditions of temperature gradient and solidification rate which are significantly different from those observed in all of the low-melting point alloys. In the CPTi case, the dendritic region is enlarged possibly due to the absence of any nucleating agent in the liquid titanium, so that the interface may supercool to a greater degree than in the more conventional alloys.

The segregation-sensitive alloys of titanium exhibit some of the features seen in steels and nickel-base alloys, such as freckles and tree rings, but to date no white spots have been seen, possibly because of the greater temperatures and larger pool volumes. The alloys are made under conditions which are intended to produce a columnar dendritic structure in order to minimize any segregation-driven defects but this aim is extremely difficult to accomplish whilst still maintaining the requisite high temperatures for surface quality.

The practical balance between these two factors usually results in a significant yield loss substantially contributing to the production cost of the alloys. As is the case for the low-melting point alloys, the prediction of the alloy structures in relation to freckle initiation is hampered by the small density difference required to drive the freckle channel as compared with the precision of our knowledge of the liquid alloy physical properties. In spite of this problem it appears that the liquid forming the freckle channel defect in titanium alloys originates at a fraction solid of approximately 0.8 [39] which is not in agreement with the present concept of the immobilization temperature, nor with the present descriptions of liquid percolation through the dendrite network. The latter indicate that under the resistance to flow existing at a fraction solid of 0.8, no flow should take place. Nonetheless, careful analysis of the freckle composition



and of the progressively segregated interdendritic liquid confirm the freckle liquid origin.

CONCLUSION

The present state of solidification modelling in the remelting processes is advanced and provides a good representation of the solidification process. However, the model output in all cases is very sensitive to the accuracy of the input parameters required. It is clear from this survey that the physical property data available to the modeler is not sufficient to permit the full power of the modeling process to be brought to bear on the actual industrial operation. It is to be anticipated that as and when such data become available the models may be used with considerable advantage in developing the processes further.

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ELECTROSLAG MELTING OF HALOGENIDE OXYGEN-FREE FLUXES*

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It is proposed to perform melting of halogenide oxygen-free fluxes by electroslag method in a copper water-cooled mould with application of a non-consumable titanium electrode. It is shown that forced rotation of the pool melt by means of a longitudinal external magnetic field intensifies melting of the charge and allows increasing productivity of the flux melting process.

Keywords: *electroslag melting, flux, slag, non-consumable electrode, magnetic field*

In electroslag welding and melting of titanium and titanium-base alloys oxygen-free halogenide fluxes (slag) [1–4] are used, basis of which constitutes CaF_2 . Fluorides and chlorides of alkaline and alkaline-earth metals are used as additions, which enable improvement of various technological and metallurgical properties of the flux (Table 1), whereby one of the main requirements to fluxes for electroslag welding (ESW) and electroslag remelting (ESR) of titanium is complete absence of oxides, because presence in flux even in small amounts of such stable oxides as Al_2O_3 , ZrO and TiO_2 does not exclude possibility of titanium contamination by oxygen [2–3].

Oxygen-free fluxes are produced by melting of the charge from initial components with their subsequent crushing up to the necessary granulation. In order to ensure maximal purity of the fluxes (first of all in relation to oxides) clean chemical components (of KhCh or Ch qualification) are used. This very condition limits possibility of melting fluxes in the flux-melting furnaces, lining of which contains oxides, that's why such fluxes are mainly produced in graphite crucibles with application of induction heating. Disadvantage of mentioned method of flux production is probability of flux contamination by carbon, which

may get into it in the process of melting from the crucible material.

Purpose of this work was investigation of possibility of producing oxygen-free fluxes by electroslag method using available modified equipment — the A-1494 installation and the TShP-10000 / 1 transformer [5]. It was proposed to perform melting of flux in a copper water-cooled mould with application of a non-consumable (non-melting) electrode.

Graphite or water-cooled electrodes with the working part, made from different refractory metals, are used as non-consumable electrodes in electroslag melting [6].

Disadvantage of the graphite electrode concerning flux melting is possibility of contamination of the latter by carbon, and of water-cooled electrodes — a relatively high cost of their manufacturing.

In addition to mentioned electrodes a titanium electrode may be used in melting of oxygen-free fluxes as a non-consumable one. Melting point of titanium is 1660°C , and of calcium fluoride (the most refractory component of the flux) — 1410°C , whereby melting point of multicomponent salt systems is, as a rule, lower [4]. So, under certain electrical conditions of melting (first of all at the slag pool voltage) one may ensure a stable electroslag process without melting of the titanium electrode. For example, in

Table 1. Chemical composition (wt.%) of fluxes for ESW (ESR) of titanium and its alloys

Flux grade	CaF_2	CaCl_2	SrCl_2	BaF_2	MgF_2	CaCl	KCl	CsI	K_2TiF_6
ÀN-02	100	—	—	—	—	—	—	—	—
ÀN-04	85	—	15.0	—	—	—	—	—	—
ÀN-06	85	15.0	—	—	—	—	—	—	—
ÀN-08	85	14.5	—	—	—	0.5	—	—	—
ÀN-010	13	—	—	73.0	13.5	—	0.5	—	—
ÀN-012	95	—	—	—	—	—	—	—	5.0
ÀN-014	95	—	—	—	—	—	—	5.0	—

*Engineers D.A. Petrov and A.M. Belov participated in the work.

**Table 2.** Conditions of AN-T4 flux melting

Slag pool voltage, V	Slag pool depth, mm	$I_{b,p}$, A	I_m , A	I_{gen} , A
10	40	1500	180	1680
10	80	650	400	1050
12	120	400	780	1180
14	160	250	1000	1250
15	200	100	1500	1600

remelting of the titanium electrode using the AN-T4 flux, when the pool voltage reduces down to 6–14 V, the electrode practically does not flash off, whereby a stable slag pool is maintained. This phenomenon was, in particular, used in welding up of a shrinkage cavity [5].

In connection with the above mentioned facts it was proposed to perform melting of the flux with application of a non-consumable titanium electrode.

Investigated AN-T4 flux was melted in a copper water-cooled mould of 140 mm diameter (Figure 1). A non-consumable electrode of 60 mm diameter was made from the VT1 commercial titanium. Calcium fluoride and strontium chloride of Ch qualification were used as initial components. General mass of the charge per one melt was 10 kg. For determining character of the electric current passage in the slag pool (flux melt) the bottom plate was electrically insulated from the mould. For additional protection of the slag pool and the heated up to high temperatures electrode against interaction with atmospheric gases local shielding of the melting space by the inert gas (argon) was performed.

Induction of the slag pool was performed with application of a «cold start» at the pool voltage 38 V. Practically at once after beginning of stable electrical current flow in the circuit, voltage was reduced down to 10 V, and further induction of the slag pool was performed. After melting of first portion of charge of 2 kg mass continuous filling of flux components into the melt zone was performed. In order to maintain constant depth of the electrode immersion into the melt and ensure more efficient conditions of heat release in the slag pool, the electrode was lifted by means of its depth increase, whereby the pool voltage varied within 10–15 V. Conditions of the AN-T4 flux melting are presented in Table 2.

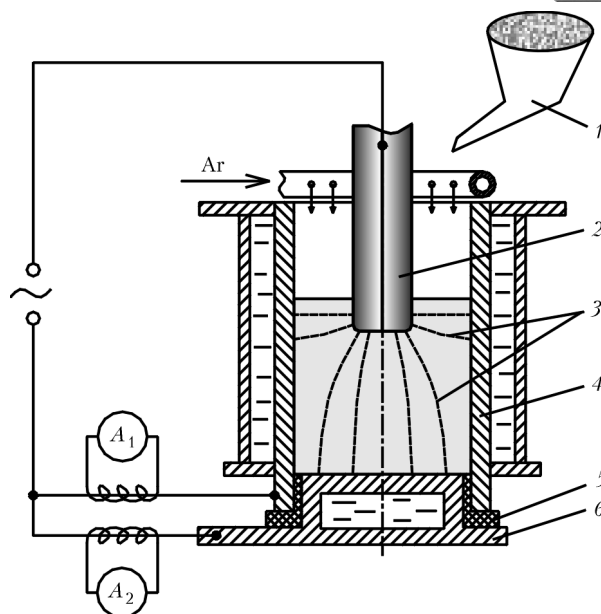


Figure 1. Scheme of electrosag melting of flux: 1 — device for filling of flux components; 2 — non-consumable titanium electrode; 3 — electric current line; 4 — mould; 5 — electric insulator; 6 — bottom plate; A_1 , A_2 — ammeters

Results of the experiments showed that at initial stage of the melting share of the mould current I_m was insignificant and constituted about 10 % (Table 2). By means of the slag pool increase the I_m share increased and at the end of melting at the pool depth 200 mm it constituted more than 90 %. This is caused by increase of the interelectrode gap (distance between end of the electrode and the bottom plate) and, accordingly, of resistance of the electrode–bottom plate circuit section. As a result general melting current I_{gen} redistributed in direction of reduction of the bottom plate current share $I_{b,p}$ and increase of the mould current share I_m .

It should be noted that flow of electric current over the mould circuit is more favorable from the viewpoint of technology, because it ensures uniform heat release in upper layers of the pool, i.e. directly in those areas into which gets the charge being remelted.

Visual inspection of the electrode end showed that in process of the flux melting at the pool voltage 10–15 V melting of the electrode practically did not occur. Just partial flashing-off of the tip, which was welded to end of the electrode for performing «cold



Figure 2. View of electrode end before (a) and after (b) melting of flux

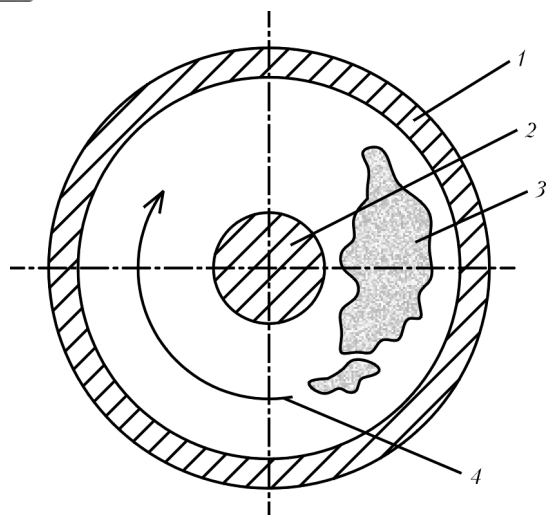


Figure 3. View of the slag pool surface: 1 — mould wall; 2 — non-consumable electrode; 3 — non-molten charge; 4 — direction of forced rotation of melt

start» of the electroslag process, was observed (Figure 2). Flashing-off occurred at initial stage at the pool voltage 38 V. Formed due to this «blintz» of titanium easily separated afterwards from general mass of the molten flux.

In the process of the flux melt its non-uniform melting occurred, which was caused by unsymmetrical in relation to the pool axis filling of the flux components into the melting space. Due to this on the pool surface on the side of the filling device a layer from the non-molten charge has formed, while on the opposite side of the mould the charge has completely melted (Figure 3).

For intensification of the charge melting and increase of the flux melting process productivity forced rotation of the melt was performed by application of the external magnetic field [7]. This did not require for development of special electromagnetic devices, because used in the magnet-controlled electroslag melting of titanium alloys moulds [5, 7] were already equipped with devices for creation in the melt zone of a longitudinal magnetic field.

Dependence of frequency of azimuthal (around the pool axis) rotation of the melt upon induction of the longitudinal magnetic field was determined experimentally (Figure 4). Frequency of the melt rotation was determined visually by frequency of rotation of the non-melted charge on surface of the pool. It was established that intensity of the melt rotation depended not just upon the magnetic field induction, but also upon ratio of currents $I_{b,p}$ and I_m . When the mould current increases, frequency of the melt rotation also increases (Figure 4). This is caused by the fact that longitudinal magnetic field interacts with radial component of the melting current, the share of which prevails in the mould current and is insignificant in the bottom plate current.

Application of forced rotation caused situation in which the charge that got on surface of the pool was entrained by the melt flows, and this enabled its more

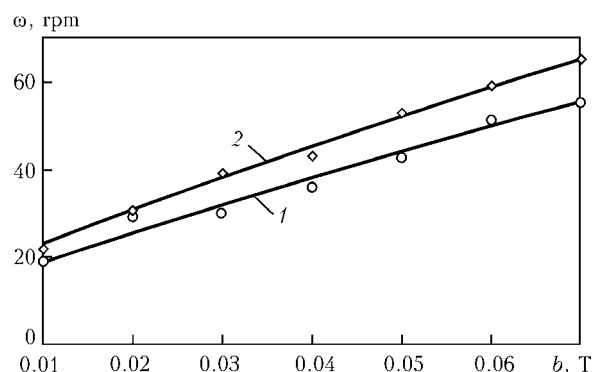


Figure 4. Dependence of frequency of melt rotation ω upon induction of longitudinal magnetic field b : 1 — $I_{b,p} = 600$ A, $I_m = 850$ A; 2 — $I_{b,p} = 450$ A, $I_m = 1000$ A

intensive melting. So, a magnetic field with induction in the melt zone 0.06 T ensured azimuthal rotation of the melt at frequency 50–60 rpm (see Figure 4), whereby maximal linear speed of rotation achieved 40 cm/s, which allowed increasing productivity of the flux melting process from 22 to 28 kg/h (by 25 %).

The above results of the investigations prove possibility of melting of the halogenide oxygen-free fluxes by electroslag method with application of a non-consumable titanium electrode. Mentioned fluxes (slag) are used in ESW (ESR) of titanium alloys, and application of additional technological equipment is not required for their production. One of the advantages of this method of flux melting, in comparison with induction melting in a graphite crucible, is absence of the flux contamination by carbon.

CONCLUSIONS

1. In electroslag melting of halogenide oxygen-free fluxes an electrode, made from the VT-1 technical titanium, may be used as a non-consumable electrode.
2. In order to prevent melting of a titanium electrode, the slag pool voltage in melting of the AN-T4 flux should not exceed 15 V.
3. Forced rotation of the pool melt by means of external longitudinal magnetic field intensifies melting of the charge and allows increasing productivity of the flux melting process by 25 %.

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ON INFLUENCE OF METAL HEATING POWER DISTRIBUTION IN MOULD IN EBCHM PROCESS ON STRUCTURE OF TITANIUM INGOTS

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Prospects of producing large-sized (more than 1 m diameter) ingots of titanium alloy using method of electron beam cold hearth melting have been substantiated. The experimentally confirmed calculation data are presented, which prove influence of temperature conditions in the mould on structure of the ingots. Technological conditions, which enable achievement of improved structure of titanium ingots, have been established.

Keywords: titanium, electron beam melting, large-sized ingots, solidification, mathematical modeling, layer-by-layer formation of ingot

At present wide application of titanium and its alloys in different fields of human activity is limited to a great degree by its cost. However, if one takes into account the fact that corrosion resistance of titanium exceeds that of stainless steels approximately 100 times (and respectively increases service life of items from titanium), dearness of titanium would not seem so high. If, in addition to this, we take into account its relatively low (4500 kg/m^3) density, than to the factors, which exert positive influence on economic characteristics of items from titanium, its specific characteristics will be added, which will cause reduction of fuel consumption, volume of bearing structures, and increase of their useful mass. At the same time for successful introduction of titanium its production cost should be reduced. One of the ways of the production cost reduction of titanium ingots is increase of their size, which allows reducing specific expenses per 1 kg of the metal due to reduction of time for technological operations, carried out for preparation for melting and after the latter, specific surface of an ingot (which allows reducing loss of the metal in machining after strain under conditions of heating outside vacuum), losses of crops ends from head and bottom parts of an ingot and edges of semi-manufactured products, and due to increase of number of standard (according to the mass) billets for their subsequent strain, which may be produced from one ingot.

Increase of size of the ingots means increase of the strain degree in production of a sort billet, which positively effects structure of a forged metal. It should be also noted that big ingots are also necessary for production of massive parts in case if welding is impossible or inadmissible because of technical requirements.

Problem of production of big ingots consists in ensuring of the metal quality. Scale factor exerts sig-

nificant influence on structure and properties of the ingot metal, frequently establishing an insuperable barrier on the way of their enlargement. As noted authors of [1, 2], for certain metallurgical technologies exist critical sizes of ingots of some alloys, above which it is impossible to produce ingots of a satisfactory quality. Professor Mitchell [2] connects this limitation with segregation and formation of big inclusions in ingots of big diameter. Main source of such «nuisances» is presence of a developed melt pool in head part of the ingot being formed. In combinations with high gradients of temperatures, characteristic of remelting processes in a water-cooled mould, a deep melt pool causes significant segregation of chemical elements. So, in order to produce a quality ingot of big (about 1 m) diameter, it is necessary to reduce volume of the melt pool. This may be done in two ways: by reduction of supplied to the mould electric power or productivity of a melt. In processes with a dependent source of heating (for example, vacuum-arc (VAR) or electroslog (ESR) remelting) these two factors are interconnected. By reducing the current we reduce productivity of the process. The problem consists in the fact that in case of reduction of current below a certain critical value at the ingot periphery near water-cooled walls of the mould, the metal stays in a solid state and will not fuse into an integral ingot (spontaneous slots and holes will occur), while it is not possible to redistribute heating in direction of peripheral part of the ingot in mentioned processes. In this case the melt pool will have a view, shown in Figure 1, *a* and *b*, and it will be impossible to significantly reduce its volume without damage to quality of the ingots.

Much greater possibilities, from the viewpoint of the melt pool depth control, represent processes of special metallurgy, which use independent sources of heating — plasma and an electron beam. By means of them it is possible not just to change general power of the ingot heating in a mould, but also efficiently redistribute heating over its surface, while application

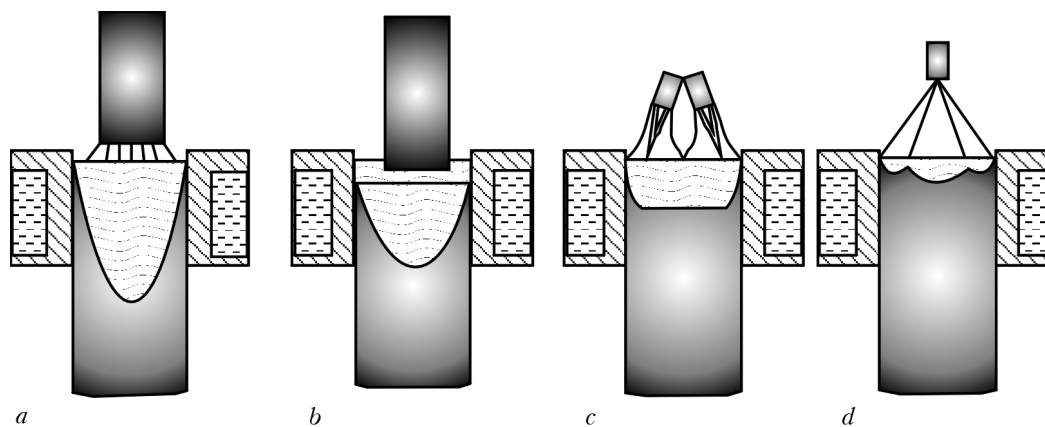


Figure 1. Schemes of ingot solidification in VAR (a), ESR (b), PAR (c) and EBR (d) processes

in mentioned methods of an intermediate unit will allow changing productivity of melting (speed of feeding of an overheated molten metal into the mould). By changing heat energy flows into the mould (amount of overheated metal fed per unit of time from the intermediate unit, and direct surface heating of the melt pool) one may change within wide range depth and configuration of the melt pool.

One manages to use redistribution of heating into peripheral part of the mould in case of plasma-arc cold hearth remelting (PAR) (Figure 1, c) to a smaller degree. This is connected with the fact that effective diameter of the heating spot on the pool surface is commensurable with dimensions of a mould. Increase of diameter of the ingots causes increase of power of the plasmatrons, which causes enlargement of the plasma jet and size of the heating spot.

The greatest possibilities for regulation of temperature conditions for formation of ingots in a mold are ensured by electron beam cold hearth remelting (EBCHR) (Figure 1, d) due to size of the heating spot on surface of a metal being heated, so called focal spot [3], and non-inertance of heating by an electron beam. For state-of-the-art melting guns of axial type, for example, «Paton-300» [4], at acceleration voltage 30 kV and beam current 10 A, diameter of the focal spot equals 20–30 mm. In comparison with diameter of the mould it constitutes just several percent, that's why diameter of the focal spot may be considered point-like. Non-inertance of heating makes it possible to transfer the beam practically instantly into the necessary point. In practice if it is necessary to heat area of a certain configuration, the beam is scanned over the preset trajectory, whereby frequency of scanning achieves 1000 Hz. So, time of movement of the beam from one point of heating to another is about 0.001 s.

It follows from mentioned above that practically any configuration of the ingot surface heating may be created by the electron beam in a mould. It is evident that for compensation of increased heat dissipation in the area of high temperature gradients near the mould walls redistribution of the electron beam heating into peripheral part of the ingot is necessary. For more objective quality and quantity assessment of such re-

distribution it is necessary to carry out numeric calculations. For investigation of thermophysical processes, proceeding during formation of an ingot, mathematical models were used [5, 6] — a 2D one for heat transfer in cylindrical ingots and a 3D one for an ingot of rectangular section. The models allow obtaining evolutionary picture of the ingot temperature field, the melt pool configuration, and zone of solid-molten state of the metal depending upon technological parameters of EBR, productivity of the process, periodicity of the melt pouring into the mould, and power of the electron beam heating.

Calculations, carried out for an ingot of 200 mm diameter and an ingot-slab of rectangular section 150 × 500 mm of the Grade 5 ASTM (Ti-6Al-4V) titanium alloy, showed that depending upon power and configuration of the ingot surface heating in the mould one may reach conditions of melting with both presence of the melt pool in upper part of the ingot and with a solid-molten state of its upper end (Figure 2). For reduction of the melt pool depth and subsequent transition to second mode it is required to reduce at constant productivity of melting general power of the metal heating in the mould and redistribute heat into peripheral part of the ingot. As showed experimentally confirmed results of the calculations, for transition to the mode with solid-molten state of the ingot end, having a rectangular section, power of heating should be less by 25 %, and in an ingot of 200 mm diameter — more than 2 times [5, 7], whereby electron beams actually heated the ingot «into a line» in its peripheral part, and central part of the ingot was heated only due to dissipated electrons (a part of the Gaussian distribution curve that exceeds diameter of the focal spot).

On basis of data on changing in time temperature field in the ingot calculations were also carried out of temperature gradient and cooling rate of the melt in each point of the ingot at the time of passage by this point of the solidus–liquidus interval, i.e. at the time of the metal solidification. Analysis of the results obtained allowed establishing dependence of values of both thermophysical characteristics upon conditions of electron beam heating of the ingot-slab. By means of reduction of the electron beam heating power

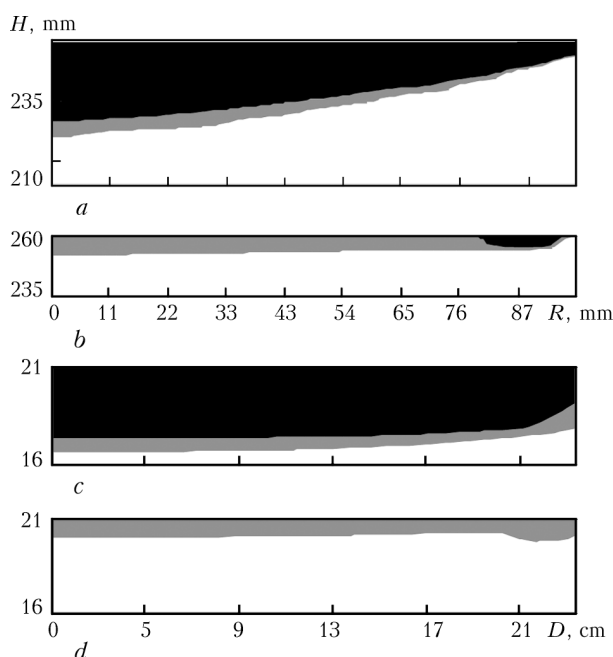


Figure 2. Temperature fields in longitudinal sections of Ti-6Al-4V titanium alloy ingots at power of electron beam heating of 35 (a) and 15 (b) kV (ingot of 200 mm diameter), 60 (c) and 45 (d) kV (ingot-slab of rectangular section 150×500 mm): R — radius of ingot; D — distance from ingot axis; H — height from bottom of ingot to its surface; black area — molten metal (> 1898 K); grey area — zone of two-phase state (1868 – 1898 K)

and transition of its maximum into peripheral part of the ingot, values of mentioned parameters increase. At the same time field of the cooling rates reflect most precisely conditions of the ingot solidification. It was established that by means of the cooling rate increase above a certain critical value (for the Ti-6Al-4V alloy it equaled about 1 K / s) a structure, completely consisting of equiaxial grains, formed in the ingots (Figure 3, b, d). Calculated temperature gradients and cooling rates also corresponded to transition from the columnar to the equiaxial structure on the Ti-6Al-4V alloy solidification map [6].

If to extrapolate obtained dependences on the process of production of large-sized ingots by the method of electron beam cold hearth melting (EBCHM), one may recommend the following technological approach:

- to redistribute power of electron beam heating into peripheral part of the ingot («line» heating) and reduce power of heating up to the value, at which

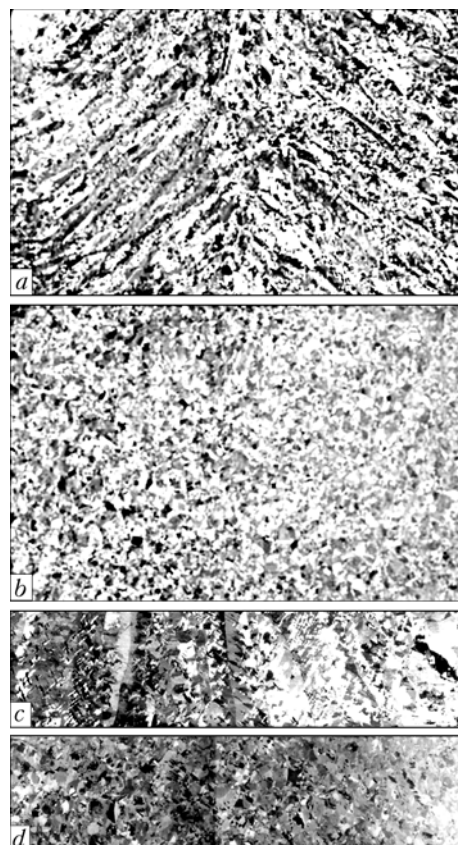


Figure 3. Macrostructure of longitudinal section of Ti-6Al-4V alloy ingots: a, b — ingot of 200 mm diameter; c, d — ingot-slab of rectangular section 150×500 mm

presence of the melt pool on periphery of the ingot is ensured;

- to ensure uniform heating of central part of the ingot at minimal level, necessary for maintaining it in molten or solid-molten state.

In practice such approach is connected with certain difficulties. So, in case of a section (diameter) increase of the EBCHM ingot, scale factor starts to affect processes of its solidification. Evidently, due to removal of central part of the ingot from cooling surface of the mould process of heat removal from the latter decelerates, and reduction of the heating power does not cause significant reduction of the pool depth. In this connection within the framework of mentioned mathematical model optimization of melting conditions of cylindrical ingots of 600 – 1200 mm diameter was performed by change of the melting productivity

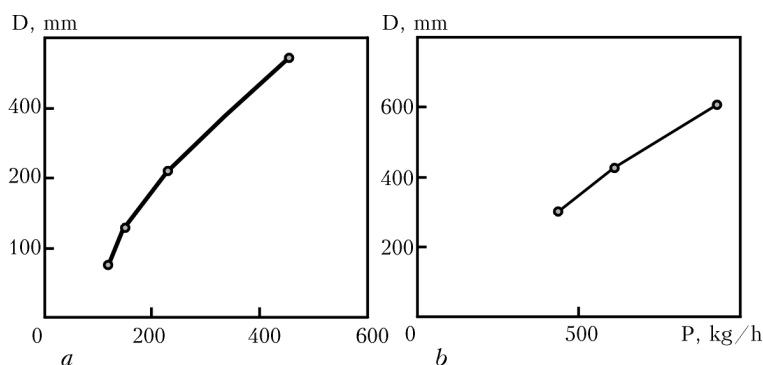


Figure 4. Influence of melting productivity P on pool depth D in ingots of 600 (a) and 1200 (b) mm diameter

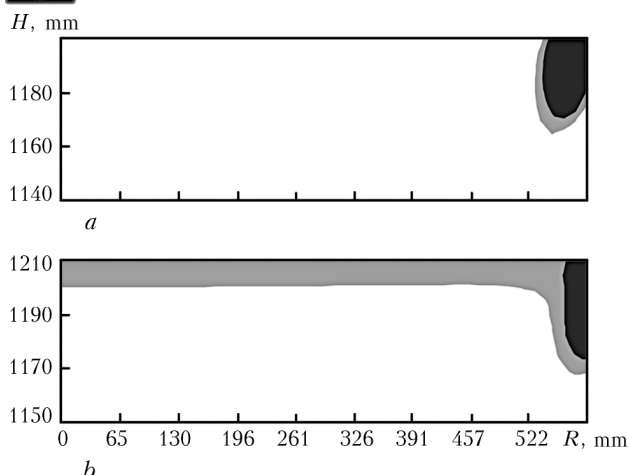


Figure 5. Temperature fields of ingot of 1200 mm diameter under conditions of layer-by-layer solidification before (a) pouring of batch and in 15 s after it (b)

(Figure 4). Radius of the ingot was chosen as a generally accepted formal criterion of the melt pool depth for assessing sufficiency of the solidification conditions. It was established that the melt pool depth did not exceed radius of the ingot at the following melt productivity: at diameter 600 mm — 350 kg / h; 840 mm — 550 kg / h; 1200 mm — 800 kg / h. Further reduction of the melt pool depth required for reduction of the melt productivity below mentioned values, which is technologically inexpedient, because in this case significantly increase time of melting and electric energy consumption per melting of 1 kg ingot.

Solution of this situation may consist, in our opinion, in reduction of central heating power of ingots of big diameter and conversion of their surface into solid state; surface temperature should always be at

the level not lower than 100 K from the melt point, whereby on periphery of the ingot surface in the mould a melt pool is maintained for forming satisfactory surface of the ingot. Such conditions will ensure metallurgical interaction between batches of the metal, poured into the mould and solidified with formation of an ingot, and simultaneously exclude segregation of the alloying elements under conditions of the melt pool absence in center of the ingot. Mentioned assumptions are confirmed by mathematical calculations. As one may see from Figure 5, a newly poured batch of an overheated metal flashes-off the previous one. In this way stability of the structure, chemical composition, and properties of the metal in the ingot are ensured, and lamination of its metal and formation of spontaneous slots and holes in it are excluded. Doubtless, idea of layer-by-layer formation of an ingot in EBCHM requires for practical confirmation by performance of experimental melts.

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TECHNOLOGY AND EQUIPMENT FOR REFINING OF MASTER ALLOYS

Aluminium-thermic reduction from oxides is a traditional technology for production of many types of complex-composition master alloys. Spontaneous occurrence of oxidation-reduction reactions with reduction of metals is caused by high chemical affinity of aluminium for oxygen. Ingots of master alloys produced by this method have such drawbacks as liquation of main elements, contamination of metal with non-metallic inclusions and increased oxygen content. These are the causes of rejection of up to 30 % of master alloys.

The E.O. Paton Electric Welding Institute developed the technology for refining vanadium-containing master alloys (up to 55 % V), based on induction melting in a sectional mould. The technology allows recycling of all wastes of master alloys.

The pilot unit was built for refining of wastes of vanadium-containing master alloys. The unit provides high-quality ingots with a diameter of up to 200 mm and weight of up to 100 kg. Annual output of the unit is up to 50 t.

The developed technology provides for remelting of lumpy scrap in a sectional mould in the inert gas atmosphere. Flux CaF₂ can be used to ensure deeper refining.

Purpose and application. The developed technology and equipment are intended for refining of master alloys with a melting point of 1500–1700 °C. The technology and equipment have passed experimental-industrial verification at the Lelinabad Rare Metal Works (Tadjikistan).

State and level of development. Experimental-industrial verification.

Principal performers and developers: Shapovalov V.A., Shejko I.V., Drs of Techn. Sci.

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SOLID-PHASE REACTIONS IN HEATING OF MULTILAYER Al / Ti FOILS PRODUCED BY ELECTRON BEAM DEPOSITION METHOD

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Structural transformations proceeding in heating of thick multilayer Al / Ti foils, produced by electron beam layer-by-layer deposition of components from the vapor phase, have been studied using methods of differential thermal analysis, X-ray diffraction methods, and scanning electron microscopy. By means of temperature increase from 350 to 650 °C successive formation of intermetallic compounds of Al–Ti system, enriched with titanium, occurs in the foil. At all stages of transformation (up to 650 °C) structure of the foil is heterophase one.

Keywords: *electron beam deposition, multilayer foil; phase transformation*

Interest to the processes, occurring in multilayer systems, which consist of alternated layers of titanium and aluminium, is stipulated by possibility of their use as protection coatings [1–3], materials for welding [4, 5], components for production of composites, etc. [6–11]. In majority of cases investigations of structural transformations at interface of titanium and aluminium alloys, which cause formation of intermetallic phases, were fulfilled on laminated structures, consisting of separate titanium and aluminium foils, subjected to machining and heat treatment [6–16]. Rather big number of works is devoted to investigation of phase transformations in multilayer Ti / Al foils, produced by methods of magnetron spraying [4, 5, 17–23].

Results of carried out investigations showed that irrespective of type of the objects (laminated or deposited multilayer structures) heating initiates processes of the reaction diffusion of atoms of components, due to which at interfaces of aluminium and titanium layers form compounds of Al–Ti system, whereby in majority of cases first and only forming phase is Al_3Ti compound [7–12, 17]. Some researches state that in case of temperature or time of annealing increase of laminated structures [13, 15] and multilayer foils, produced by magnetron spraying [18], subsequent transformation causes formation of the AlTi compound.

Authors of [19, 20] have established that sequence of formation of phases in multilayer foils, produced by magnetron spraying, may be affected by period of alternation of the layers (total thickness of one layer of aluminium and one layer of titanium). So, heating of foils with period up to 200 nm causes direct formation of the AlTi compound in foils with periods 500 and 1000 nm, which is preceded by formation of intermediate Al_3Ti and Al_2Ti phases. Single-stage process of formation of the AlTi phase was also ob-

served when period of alternation of the layers was 5 nm [21]. In case of the period increase up to 500 nm formation of the AlTi phase occurred through intermediate phases, when in a specimen the Al_3Ti and Ti_3Al phases were simultaneously detected.

As showed investigations of other multilayer systems, for example Al / Ni foils [24, 25], one of the factors, which determine kinetics of reaction during heating, is structure of the layers (first of all size of the grains). As far as in deposited laminated structures mean size of the grains and thickness of a layer are approximately equal, observed difference in reaction processes, occurring in Al / Ti foils with small and big periods of layers [19, 20], may be stipulated by different microstructure of separate layers. It is known that characteristics of the material structure depend upon conditions of deposition even in case of using the same method of production of the condensates [26, 27]. That's why it would be logical to expect change in structure of the layers and, as a result, differences in synthesis processes of intermetallics in case of using different methods of production of laminated materials.

In development of the technology for formation of laminated materials in the form of foils or coatings a number of advantages, in comparison with other methods, have electron beam evaporation and deposition in vacuum of vapors of pure elements [28]. This method ensures high (up to 100 nm / s) rates of deposition, which makes it possible to produce thick (up to 200 μm) foils within short time, and control with high accuracy ratio of chemical elements in the foil.

Taken into account stated above, the actual issue is establishment of consequence of phase and structural transformations in the multilayer Al / Ti foils, produced by deposition of vapor component phases. These results are necessary, first of all, for determining heat treatment conditions of such foils, which allow forming structural states on basis of certain intermetallic compounds, and determination of influ-

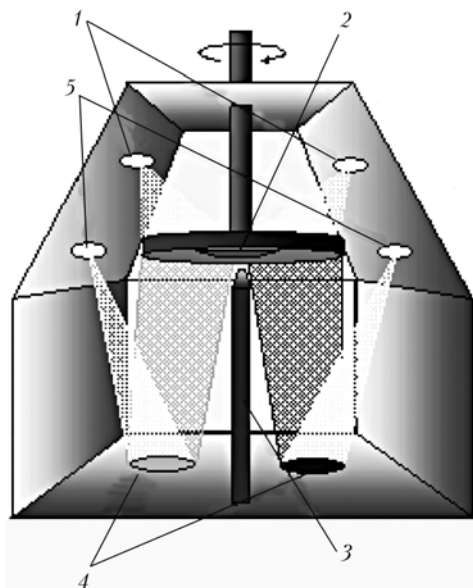


Figure 1. Scheme of installation for production of multilayer Al / Ti foils: 1 — heating guns; 2 — rotating substrate; 3 — screen; 4 — targets; 5 — evaporation guns

ence of initial structure of the multilayer foils on these processes.

In the work, using as an example multilayer Al / Ti foils of close to equiatomic composition that differ by the period of alternation of the titanium and aluminium layers, structural transformations were investigated, stipulated by continuous heating of these foils at constant rate and at different temperatures.

Methods of production and investigation of the foils. Investigated foils were produced on the installation, which is described in detail in previous works (for example, [28]). Schemes of the deposition systems are presented in Figure 1. Aluminium and titanium ingots were placed in two separate moulds, cooled by flowing water, which was located in the vacuum chamber, divided into two parts by an impermeable screen. Continuous vapor flows, produced by means of electron beam guns from aluminium and titanium ingots, were deposited on a substrate, rotating above the screen around a vertical shaft. Due to the impermeable screen vapor flows got only on that part of the substrate, which was located above a respective ingot. Rotation of the substrate ensured sequential deposition of aluminium and titanium layers on the substrate, thickness of which was determined by ratio of the vapor flow density and speed of the substrate rotation. General thickness of the foil depended upon period of the deposition process and varied from 10 to 100 μm .

Prior to deposition of the aluminium and titanium layers the CaF_2 layer was deposited on the substrate, which made it possible afterwards to easily separate foil from the substrate. Pressure in the chamber during deposition was maintained at the level below $5 \cdot 10^{-3}$ Pa. Temperature of the substrate in the process of deposition was 170–200 $^{\circ}\text{C}$. Intensity of evaporation ensured rate of deposition 50 nm / s.

For characterizing changes, which occurred in specimens of foils during heating, methods of differential thermal analysis (DTA), scanning electron microscopy (SEM), and X-ray diffraction were used.

Investigations by the DTA method were carried out on the VDTA-8 [29] installation, whereby so called method with a diluent was used [30], in which copper foil of 100 μm thickness was used as the latter. From the deposited Al / Ti foil specimens of 10×10 mm size were cut out, which were placed between copper plates. This package was heated in the DTA installation in helium up to the preset temperature at a constant rate 50 $^{\circ}\text{C}$ / min.

After measurements by the DTA method, carried out for one of the foil specimens in initial state, other specimens were heated under the same conditions in the installation for DTA up to several selected temperatures; if necessary they were soaked at these temperatures within a preset time and then cooled down to room temperature. Annealed specimens were investigated by the X-ray diffraction and SEM methods.

X-ray diffraction measurements were carried out in standard θ – 2θ geometry (θ is the diffraction angle) on the DRON-4 diffractometer in $\text{CuK}\alpha$ radiation. Specimens for investigation of the initial state represented pieces of foil, which were milled after heat treatment up to powder-like state. Textured state of layers in the deposited foils was investigated using the DRON-2UM diffractometer, equipped with special attachment.

Microstructure of the specimens was studied using the scanning electron microscope CanScan-4. In order to increase descriptiveness of the microstructure analysis different reactivities were used for chemical etching of the foil cross sections, which ensured primary etching of structural components, enriched by aluminium or titanium. For determining chemical composition of the specimens the microscope was equipped with the energy dispersion spectrometer Energy200.

Experimental results. Structure of Al/Ti foils in initial state. In the work the foils were investigated with alternation period of layers from 40 to 1 μm . General thickness of the foils was 50–70 μm .

Results, obtained in process of the investigation, are illustrated using as an example two foils, which significantly differ by thickness of aluminium and titanium layers. The foil that has mean chemical composition 56 at.% Al–44 at.% Ti consists of aluminium layers of 0.44 μm thickness and titanium layers of 0.37 μm thickness; general thickness of this foil is 67 μm . Another foil of mean chemical composition 61 at.% Al–39 at.% Ti consists of aluminium and titanium layers of respectively 0.026 and 0.018 μm thickness; general thickness of the foil is 52 μm . For briefness of presentation these two foils will be further designated as F1 (a microlayer one) and F2 (a nanolayer one). If one follows equilibrium phase diagram Ti–Al (Figure 2), mean chemical composition of F1



corresponds to the area of the TiAl compound formation, and F2 — two-phase TiAl + Al₂Ti area.

In Figure 3 typical microstructure of cross section of the foil specimens in initial state is presented. One may see from the Figure that the foils consist of continuous relatively even layers. Even if mixing at boundary of the layers is present, it is insignificant. In Figure 3, *b* boundaries of columnar multilayer grains of about 0.5 μm thickness, oriented perpendicular to the layers, are observed. Such columnar structure is also characteristic of the F1 specimen. On microstructure of this specimen (Figure 3, *a*) columnar elements of the structure are not manifested, because their thicknesses constitute on average 1.5–2.0 μm. Length of these columnar grains may be different; it may also reach thickness of the foil.

On X-ray diffraction images, obtained for the foil specimens in initial state, only the lines corresponding to α-Ti and Al (Figure 4, *a*) are present. It should be noted that relative intensities of diffraction lines both for α-Ti and Al prove textured state of the layers. Results of a special X-ray diffraction investigation confirmed presence of texture in the aluminium and titanium layers and made it possible to determine its characteristics. In Figure 4, *b* distribution of density of poles (001)Ti + (111)Al and (103)Ti is shown. These results indicated axial type of the texture, whereby orientation ratio (001)Ti ∥ (111)Al is fulfilled. So, in the process of deposition atomic planes of aluminium and titanium with close packing are arranged parallel to each other in the foil plane. It should be noted that the same type of texture was

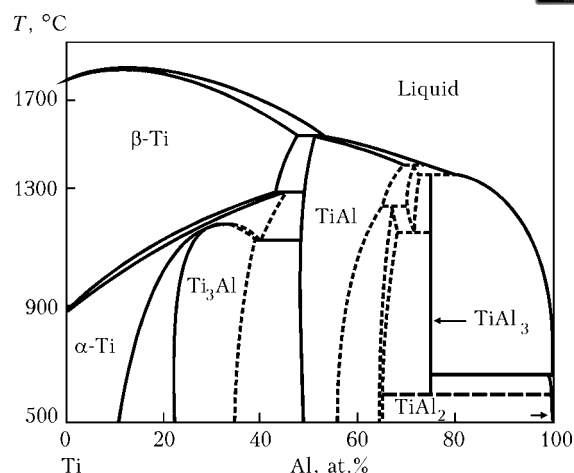


Figure 2. Phase diagram of Ti–Al system [31]

earlier established on basis of X-ray diffraction images of specimens in the form of foils in powder-like state from thin multilayer Al/Ti films, produced by the method of magnetron spraying [7].

Structural transformations stipulated by slow heating. In Figure 5 the results are presented, which characterize heat release processes occurring in foils during their heating at constant rate. One may see from the Figure that in the process of heating exothermal phase transformations occur in the foils. It should be noted that heat patterns of foil specimens with different thickness of layers differ. Heat pattern of the F1 foil specimen with thick layers (Figure 5, *a*) represents wide peak of a complex shape; phase transformation starts approximately at 400 °C. Presence of flexes on the heat pattern proves simultaneous pro-

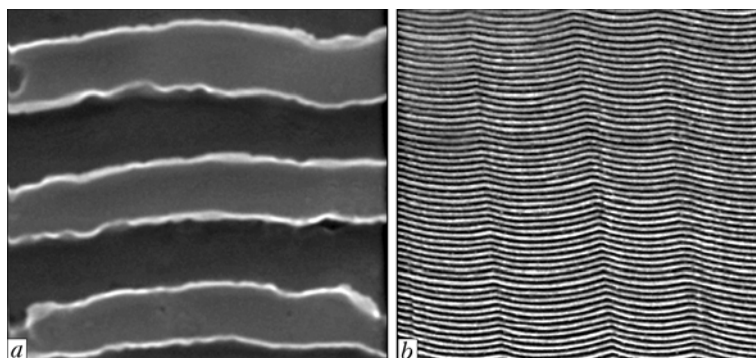


Figure 3. Microstructure of cross section of F1 (*a*) and F2 (*b*) foil specimens after deposition: light strips — titanium layers; dark strips — aluminium layers (×25000)

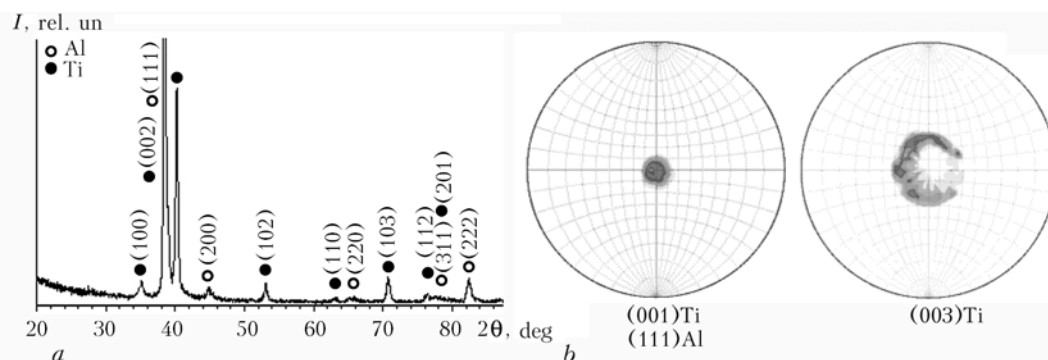


Figure 4. X-ray diffraction image (*a*) and distribution of density of poles (001)Ti + (111)Al and (103)Ti (*b*) in F1 foil specimens after deposition: *I* — intensity

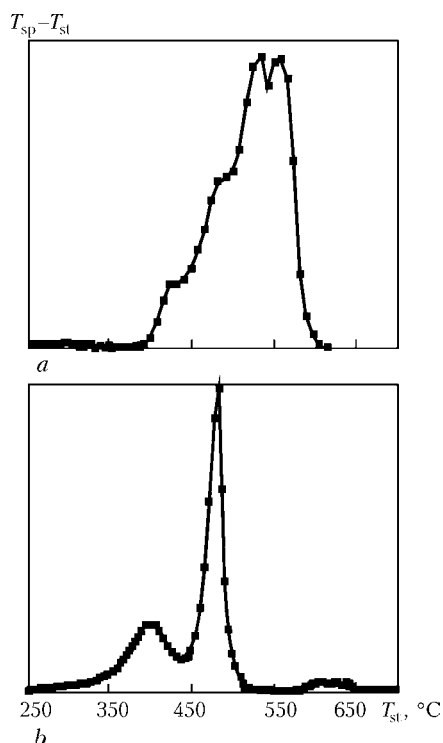


Figure 5. DTA curves of F1 (a) and F2 (b) foil specimens obtained in heating at rate 50 °C / min: T_{sp} , T_{st} — temperature of specimen and standard, respectively

gress of several solid-phase reactions. During heating of the F2 foil with thin layers (Figure 5, b) thermal peaks on the heat pattern are divided, and thermal effects have a clearly pronounced character. Temperature intervals of the transformations constitute 300–400, 440–480, 550–610 °C.

So, it follows from DTA results that during heating of multilayer Ti / Al foils up to 600 °C at least three phase transformations occur in them, whereby temperature of beginning of the transformations depends upon the alternation period that shifts in direction of lower values by means of thickness reduction of the layers.

In order to establish sequence of phases, formed in the process of heating, the specimens, which were soaked within 5 min at 350, 450, 550 and 600 °C, and then cooled down to room temperature, were investigated. Some specimens were soaked at temperature 350 °C within 1.5, 2 and 5 h.

In Figure 6 X-ray diffraction images, obtained for the F1 specimens heated to different temperatures, are presented. On diffractogram of the specimen heated up to 350 °C (Figure 6, a) additional diffraction lines were not detected. However, they have a peculiarity — presence of a «shoulder» (in Figure 6, a it is shown by the arrow) on the side of big diffraction angles near the peak formed by superimposition of the lines (002)Ti and (111)Al. This peculiarity may be stipulated by presence of a new phase, content of which is not sufficient for manifestation of other diffraction lines. For identification of this phase other specimens of this foil, heated up to 350 °C and soaked at this temperature within 1.5 and 2 h, were investigated. At annealing within 1.5 h any changes in the X-ray diffraction image were not detected. After soaking of a specimen within 2 h at the same temperature, in addition to aluminium and titanium lines, peaks from the Al_3Ti phase were registered on the diffractogram. That's why one may assume that asymmetry of peak (002)Ti + (111)Al, characteristic of diffractograms of the specimens annealed at 350 °C within 5 and 30 min, is connected with beginning of the Al_3Ti phase formation, volume share of which increases by means of the annealing time increase. In a specimen of the same foil, soaked at the temperature 350 °C within 5 h, the Al_3Ti phase was not detected, but there were diffraction signs of the Al_5Ti_2 phase formation.

Soaking of the specimens within 5 min at 450 °C also enabled formation of the Al_5Ti_2 phase (Figure 6, b), whereby a portion of titanium and aluminium in the specimens remains untransformed, which is proved

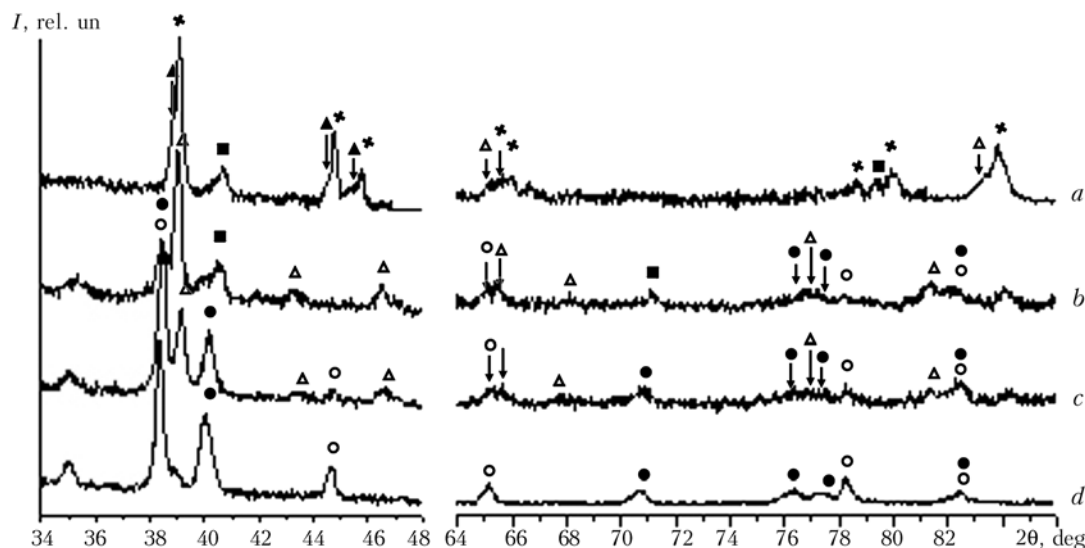


Figure 6. Characteristic fragments of diffractograms of F1 foil specimens heated at rate 50 °C / min up to $T = 600$ (a), 550 (b), 450 (c), 350 (d) °C with soaking at these temperatures within 5 min: ○ — Al; ● — Ti; Δ — Al_5Ti_2 ; × — Al_2Ti ; ▲ — AlTi; ■ — peaks, positions of which correspond to structure with hexagonal symmetry of lattice

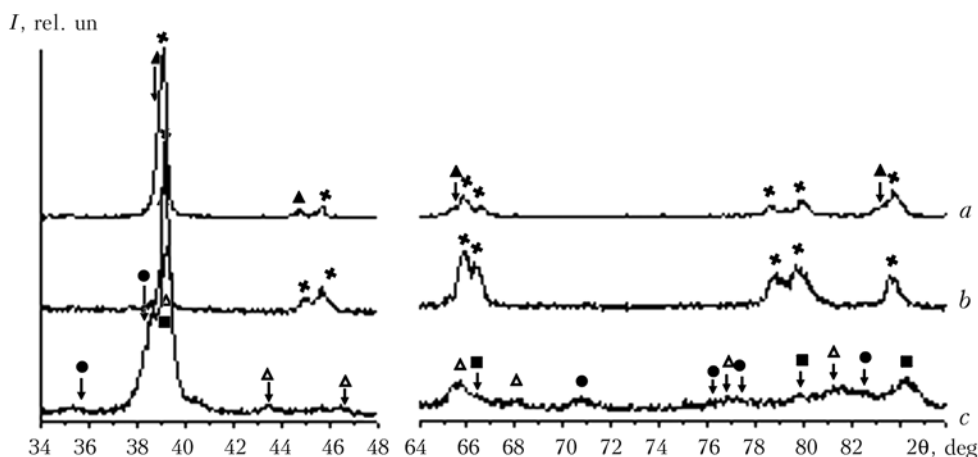


Figure 7. Characteristic fragments of diffractograms of F2 foil specimens heated at rate 50 °C / min up to $T = 600$ (a), 550 (b) and 450 (c) °C with soaking at these temperatures within 5 min: ● — Ti; ■ — Al_3Ti ; Δ — Al_5Ti_2 ; × — Al_2Ti ; ▲ — AlTi

by presence on the diffractograms of lines of these elements. At temperature increase up to 550 °C (Figure 6, c), in addition to the maximums obtained at the temperature 450 °C, additional maximums of intensity are observed, which prove formation of one more phase. These diffraction lines may be identified as the ones belonging to a structure with hexagonal symmetry of the lattice with parameters $a \approx 0.2915$ nm and $c \approx 0.465$ nm, i.e. at 550 °C, in addition to aluminium-base phases, a titanium-base phase forms, which is, evidently, represented by the AlTi_3 intermetallic of non-stoichiometric composition.

After heating of the specimen up to 600 °C (Figure 6, d) peaks from the structure with hexagonal lattice are preserved, and a system of new peaks, connected with formation of the Al_2Ti and AlTi phases, appears. On diffractogram of the specimen, heated up to 650 °C, redistribution of intensity between peaks of the Al_2Ti and AlTi phases occurred, which proved increase at this temperature of volume share of the AlTi phase due to reduction of the Al_2Ti phase content.

Similar investigations were carried out for specimens of the F2 foil, alternation period of layers in which is significantly lower than in the F1 foil (0.044 μm in comparison with 0.810 μm). X-ray diffraction images from specimens of the F2 foil, heated up to temperatures 450, 550 and 600 °C, are presented in Figure 7, from which one may see that similar to specimens of the F1 foil in these specimens at increase of temperature sequential formation of Al_3Ti , Al_5Ti_2 , Al_2Ti and AlTi phases, is observed. However, formation of each of the phases in these specimens occurs at a significantly lower temperature than in specimens of the F1 foil. This result correlates with DTA data, which prove that temperature of beginning of the transformations in the F2 foil, in comparison with the F1 foil, has lower values (see Figure 5). In addition, signs of formation of the phase with hexagonal symmetry of the lattice, which is present in specimens of the F1 foil heated up to 550 °C and more, were not registered.

Specimens for metallographic investigations were prepared with application of electrolytes, which ensure selective etching of structural components enriched either by titanium or aluminium. Selective etching allowed detecting peculiarities of formation in layers of intermetallic phases. In Figure 8 changes of the foil cross section structure are shown that occur during heating of the specimen up to different temperatures and their soaking at these temperatures within 5 min. One may see that heating of the foil up to the temperature 350–400 °C activates mutual diffusion between components of the layers, which causes change of their structure (Figure 8, a, b). So, in titanium layers «bridges» appear, phase contrast of which proves that they are enriched by aluminium (Figure 8, b, c, zone A) in comparison with bulk of the layer. Analysis of phase contrast on microstructures of the specimens, subjected to selective etching for presence of aluminium and titanium, allows assuming that the «bridges» correspond to grain boundaries in titanium layers, within which titanium-base phases are formed due to surface diffusion, for example, AlTi_3 or solid solution Ti–Al (Figure 8, c, zone B). This assumption is confirmed by presence on diffractograms of the foil, annealed at temperature more than 550 °C, of the peaks, position of which corresponds to the phase with hexagonal lattice. By means of temperature increase of the specimens up to 550 °C, thickness of titanium interlayers reduces and a composite is formed, consisting of a mixture of intermetallic phases enriched with titanium and aluminium (phase contrast in Figure 8, d).

Discussion of results. So, during heating of multilayer Al / Ti foils with approximately equiatomic ratio of components within temperature range 250–650 °C succession of phase transformations is observed in the system, which schematically may be presented in the form $\text{Al}_3\text{Ti} \rightarrow \text{Al}_5\text{Ti}_2 \rightarrow \text{Al}_2\text{Ti} \rightarrow \text{AlTi}$. In addition, during heating approximately up to temperature 550 °C Ti_3Al intermetallic is formed in the microlayer foil, which preserves in the specimens during further increase of temperature. In the nanolayer foil

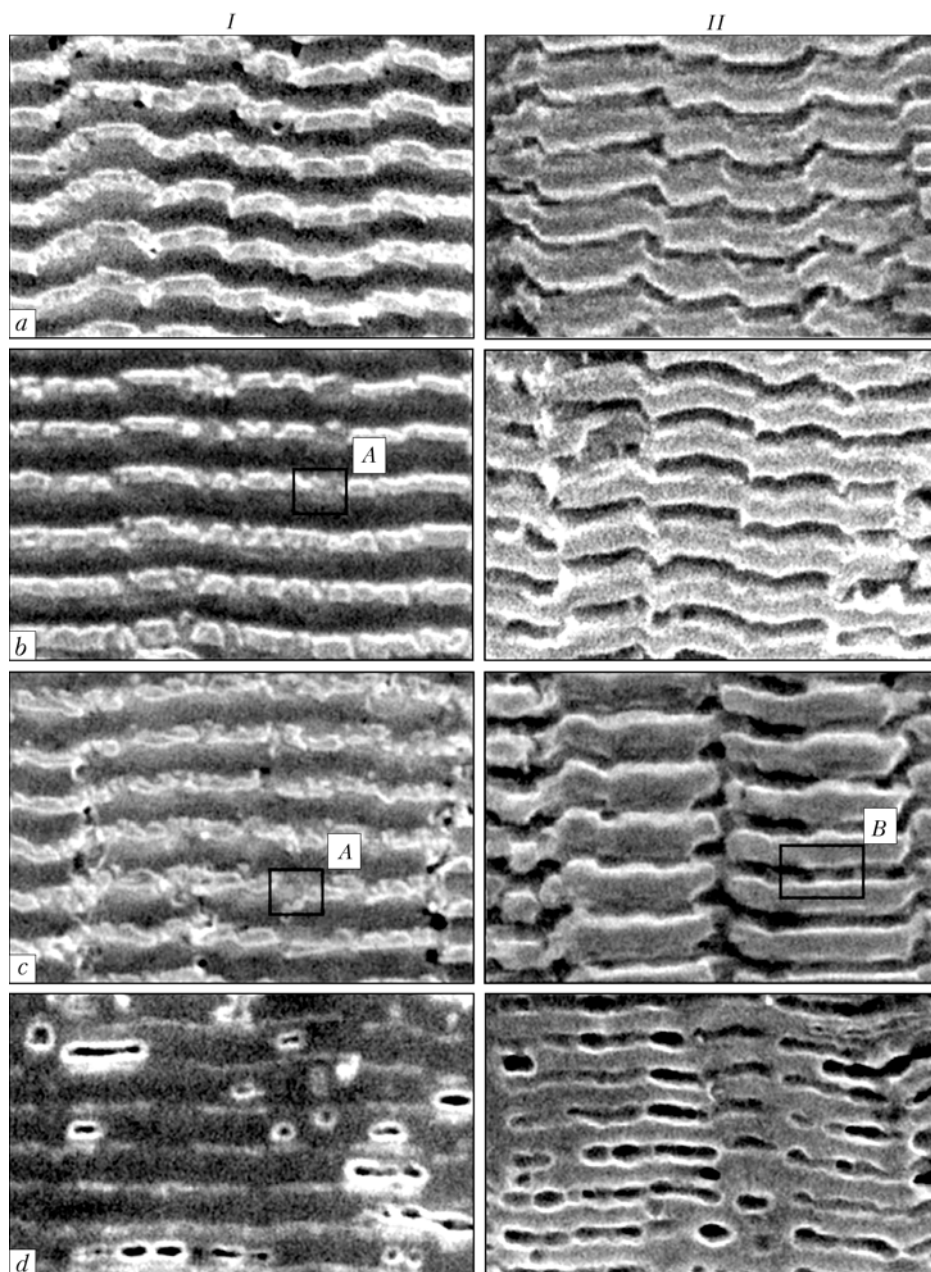


Figure 8. Microstructure of cross section of F1 foil specimens in initial state (a) and heated up to 350 (b), 450 (c) and 550 (d) °C with soaking at these temperatures within 5 min: I–II — primary etching of aluminium and titanium respectively ($\times 7000$)

diffraction signs of Ti_3Al intermetallic formation are absent.

Formation at first stage only of Al_3Ti phase is not unexpected, because such phenomenon was previously observed in investigations of Ti–Al laminated systems. Formation of Al_3Ti as the first product of the reaction diffusion process in Al–Ti system matches also predictions of the model of efficient formation heat, suggested by Pretorius et al. [32, 33].

If one follows thermodynamic principles [13, 34] or Pretorius model, next phase after Al_3Ti should be AlTi phase. Formation of exactly this phase (but at higher temperatures) was registered at second stage of transformation in laminated Al / Ti structures [13, 15] and multilayer foils, produced by magnetron spraying [19, 20]. In our case in the foils, heated up to 450 °C, after Al_3Ti , Al_5Ti_2 compound forms (see

Figure 6, b). Within framework of existing models this result does not have explanation. Formation of both Al_5Ti_2 and Al_2Ti is possible provided preliminarily AlTi phase was formed [13, 35]. At the same time results of X-ray diffraction investigations prove that in the investigated by us foils, AlTi phase is formed either after Al_2Ti phase or simultaneously with it (see Figure 6, d).

According to Pretorius model, formation of AlTi_3 intermetallic should be the next step after formation of intermetallic AlTi. Our data prove appearance of AlTi_3 compound at lower temperature than temperature of AlTi formation (see Figure 6, c).

So, sequence of phase transformations in investigated multilayer Al / Ti foils, firstly, cardinally differs from phase transformations, established earlier for multilayer Al / Ti structures, and, secondly, may



not be explained proceeding only from principles of thermodynamics. As far as formation of intermetallics in systems of this kind is result of reaction diffusion [36], one may assume that sequence of phase transformations is determined not just by thermodynamics, but also by diffusion kinetics.

It is considered generally recognized that up to the aluminium melting point (660 °C) in the Al / Ti pair diffusion occurs only on side of aluminium and hence Al₃Ti phase may form only on side of titanium [37–41]. The «bridges» on microstructures (Figure 8) that cross titanium layers may be considered as evidence of grain-boundary diffusion of aluminium atoms into titanium layers, which enables formation on intergranular boundaries of a phase enriched by titanium, for example, AlTi₃. It is logically to assume that by means of size reduction of the grains in case of the nanolayer foil density of grain boundaries will increase, and, as a result, one may expect increase of a flow of aluminium atoms over titanium grain boundaries that should cause more intensive formation of AlTi₃ compound in comparison with microlayer foils, while in reality the opposite is observed. Diffraction signs of AlTi₃ phase formation in nanolayer foils were not detected, although investigated foils in majority of cases are enriched with titanium in comparison with microlayer ones. Observed phenomenon may be result of the fact that in nanolayer foils either AlTi₃ phase with more dispersed sizes is formed, or its formation is suppressed.

In [13, 42–44] the results were obtained that prove diffusion of titanium atoms into aluminium at a lower temperature than melting point of aluminium. So, it was established in [13] that after annealing of laminated Al / Ti structures at 575 °C thickness of aluminium layers reduced almost 6 times — this is more than reduction of thickness of titanium layers. Such result, evidently, proves mainly diffusion of titanium atoms into aluminium, because growth of intermetallic Al₃Ti layer occurs mainly on side of aluminium. It was detected in [42] in investigation of defects of vacancy type using method of positron-annihilation spectroscopy in three-layered Ti / Al / Ti structure deposited on single crystal of silicon, that main diffused element at Ti / Al interface is titanium at temperature 280 °C.

It is shown in [43, 44] that titanium layers, deposited on single crystals of aluminium, remain stable approximately up to 400 °C. At a higher temperature titanium atoms diffuse into aluminium lattice and occupy substitution nodes. That's why it would be logic to assume that in case of laminated condensates, produced by electron beam deposition, diffusion of titanium atoms occurs similar to the pattern, observed in [43, 44], i.e. due to volume diffusion.

In conclusion it should be noted that significant volume of results, obtained in works of other researchers, concerns laminated Al / Ti structures, average chemical composition of which is, like in this work, close to the equiatomic one. Nevertheless, ex-

cept very rare exclusions, during heating of the specimens in these works only Al₃Ti and AlTi phases were detected in contrast to the chain of transformations Al₃Ti → Al₅Ti₂ → Al₂Ti → AlTi, registered by us. As far as different methods were used for production of Ti / Al laminated structures (cold or hot rolling and magnetron spraying in earlier published works and electron beam deposition in this work) it would be logic to expect significant difference in microstructure of the layers and structure of the interphase surface. As far as these factors should exert significant influence on diffusion processes, exactly they may be main reason of noted differences in processes of phase transformations.

CONCLUSIONS

1. Irrespective of value of alternation period of layers in multilayer Al / Ti foils, produced by electron beam deposition, averaged chemical composition of which is close to the equiatomic one, their heating at constant rate 50 °C / min initiates sequential formation of Al₃Ti, Al₅Ti₂, Al₂Ti and AlTi phases. Temperature of formation of the phases depends upon thickness of layers in the foils, whereby by means of reduction of alternation period of the layers it shifts to lower values.

2. Formation in microlayer foil of AlTi₃ phase side by side with Al₃Ti → Al₅Ti₂ → Al₂Ti → AlTi sequence may prove realization in such structures of two channels of phase transformations, stipulated by two diffusion processes — volume diffusion of titanium atoms into aluminium, and grain boundary diffusion of aluminium atoms into titanium. Absence on the diffractograms of diffraction signs of AlTi₃ phase formation in the nanolayer foils may prove either suppression of this process or more disperse sizes of the crystallites. It is assumed that a number of additional investigations will be carried out for finding out influence of thickness of the layers on formation of AlTi₃ compound.

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INTERACTION OF STRUCTURE, MICROSCOPIC LIMIT OF ELASTICITY AND INTERNAL FRICTION OF COMPOSITE MATERIALS PRODUCED BY METHODS OF ELECTRON BEAM TECHNOLOGY

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Results of determination of microscopic limit of elasticity and characteristics of internal friction of pure metals and dispersion-strengthened and microlayer materials, produced by methods of electron beam evaporation and condensation in vacuum, are presented. Existence of non-monotonous dependence of the energy dissipation characteristics and microscopic limit of elasticity upon a structure, formed in the process of production and subsequent processing of the material, is established.

Keywords: electron beam evaporation, condensed materials, microscopic limit of elasticity, logarithmic decrement of attenuation, background of internal friction

Microscopic limit of elasticity that characterizes beginning of development of irreversible plastic changes in the material is to a significant degree determined by the dislocation structure, formed in the process of production and subsequent processing of the material. Internal friction is also most structurally sensitive to properties of materials and alloys.

In [1–4] non-monotonous dependence of microscopic limit of elasticity τ_s of a number of metal materials with different background upon level of preliminary plastic strain, produced by uniaxial tension of pure metals (copper, nickel), phase concentration in double-phase NiCr–Al₂O₃, Fe–Pb and thickness of a layer in microlayer Fe / Cu condensed materials, was established.

In this article results of investigation of interconnection of the structure, microscopic limit of elasticity and internal friction of different materials are presented.

Pure metals (copper, nickel) were produced by the method of electron beam remelting; double-phase NiCr–Al₂O₃, Fe–Pb and microlayer Fe / Cu — by method of electron beam evaporation with subsequent condensation on the metal substrate, on which ZrO₂ separating layer was applied. Methodology of producing the materials is presented in greater detail in [1–4].

Microscopic limit of elasticity τ_s was determined on basis of internal friction, measured under conditions of simultaneous action on the specimen of periodic, occurring under influence of torsion free-attenuating oscillations and monotonously increasing in time additional tangential stresses [5]. Measured internal friction δ consists of the internal friction background δ_0 (determined by movement of dislocations) and mi-

croplastic deformation energy losses δ_{pl} . Consumption of elastic energy is mainly connected with formation of new dislocations [6]: $\delta = \delta_0 + \delta_{pl}$. Values τ_s were assumed as the highest elastic stresses, at which microscopic deformation losses equal zero [5]. Initial amplitude of deformation, occurring under action of the alternating stress, did not exceed $1 \cdot 10^{-5}$ rel. unit.

Additional tangential stress was significantly lower than microscopic limit of elasticity and did not exceed $(1/3)\tau_{0.2}$. In experiments with different materials rate of additional stress increase constituted 1.23–6.00 MPa / s. Frequency of torsion oscillations varied from 0.5 to 12.8 osc / s. The experiments were carried out in amplitude-independent area at room temperature.

Structure of the specimens was studied using the JSEM-200 electron microscope [2, 7].

Influence of preliminary plastic strain on structure, microscopic limit of elasticity and internal friction. *Nickel.* Massive specimens from nickel of 99.99 % purity were annealed in vacuum at 700 °C within 1 h, than deformed by uniaxial tension at 20 °C within strain range 0–24 %. Change of values of microscopic limit of elasticity τ_s , internal friction δ and internal friction background δ_0 , depending upon preliminary plastic strain, are shown in Figure 1. It follows from [7] that at strain 2–8 % a cellular structure with mean size of a cell 1.3 μ m is formed. By means of strain increase up to 16 % reduction of the cell size from 1.3 to 0.6 μ m occurs. Evidently, at stresses, exceeding τ_s value, the longest sources of dislocations are actuated. By means of the specimen strengthening stress τ_s increases and new shorter sources are included into the work, segment length values of which meet conditions that ensure generation of dislocations. Simultaneously blocked sources may renew their work.

At $\varepsilon \approx 16$ %, τ_s has maximal value. By means of τ_s increase level of internal friction reduces and achieves minimal value at the strain, corresponding to maximal τ_s value. At mentioned strain structural

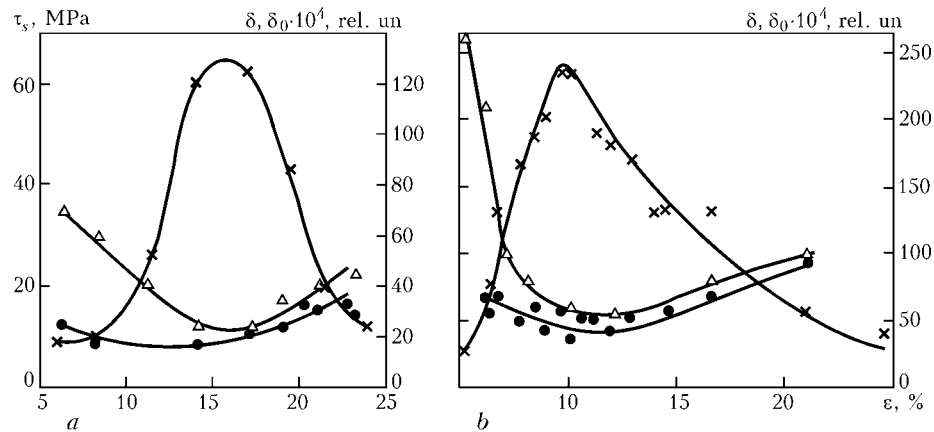


Figure 1. Dependence of τ_s , δ and δ_0 upon preliminary plastic strain of nickel (a) and copper (b); here and in Figures 2–4: Δ — δ ; $\bullet$ — δ_0 ; $\times$ — τ_s

changes were registered, connected with dynamic return: partial disintegration of cellular structure and formation of dense extended dislocation accumulations (fragments) with angular disturbance 3–5°.

The biggest width of the fragments is 0.95–1.00 μm . Mentioned processes stipulate reduction of τ_s value (Figure 1). Occurrence of continuity disturbances of different kinds, which may become concentrators of stresses and be accompanied by reduction of τ_s level in case of strain increase, is not excluded, because microplastic deformation will occur in the most overstrained microareas at lower τ_s values. These assumptions match experimental data, presented in [8–11]. So, in [8] reduction of the material density is explained by presence of microcracks, formed in the process of the austenite steel plastic strain. In [9, 10] dissipation of light on ultramicroscopic heterogeneities, formed in the process of plastic strain, was registered. Presence of microcracks was also detected in the process of autoion-microscopic investigation of structure of preliminarily strained by tension tungsten at deformations, corresponding to III stage of hardening [11].

Simultaneously with reduction of the τ_s value at deformation more than 16 % increase of internal friction δ and internal friction background δ_0 were registered. Evidently, microstructure, formed due to preliminary plastic strain, determines the internal friction background at each preset strain. So, increase of the energy dissipation occurs not just due to the microplastic deformation losses δ_{pl} , but also increase of the internal friction background.

Copper. Massive specimens from oxygen-free copper of 99.85 % purity were annealed in vacuum at 700 °C within 1 h and then subjected to preliminary tensile strain 0–35 %. Presented in Figure 1, b results of determination of τ_s , δ and δ_0 dependence upon degree of copper strain are similar to the data, obtained for nickel (see Figure 1, a).

According to results of electron-microscopic investigations [2], at deformation about 2 % cellular structure with average size of the cell 1.8–2.0 μm forms. By means of deformation increase approximately up to 8 %, size of the cell reduces down to 1.2 μm . Value

of τ_s achieves its maximal level at $\varepsilon \sim 10$ %. At deformation above 10 % partial disintegration of the cellular structure occurs, and boundaries of fragments with angular disturbance about 1° appear.

Formation of fragmentized structure is accompanied, like in nickel, by reduction of the τ_s value and increase of the internal friction background δ_0 and internal friction δ . Proceeding from the fact that microplastic deformation is mainly stipulated by propagation of dislocations and assuming that it occurs according to Frank–Reed mechanism, length of the dislocation loop, able to generate dislocation, was estimated by the τ_s value:

$$L_c = \frac{Gb}{\tau_s}, \quad (1)$$

where L_c is the dislocation source length; G is the shift module; b is the Burgers vector.

At $\varepsilon = 2$ %, $L_c = 2.05 \mu\text{m}$ that coincides with the cell size 1.8–2.0 μm . Deformation $\varepsilon = 8$ % corresponds to $L_c = 0.61 \mu\text{m}$, i.e. approximately 2 times less than average size of the cell. This is, evidently, connected with presence of significant number of dislocations in internal areas of the cells, which may act as sinks for movement of dislocations thus causing increase of τ_s and ensuring work of the sources at lower L_c values.

Similar to presented above δ_0 dependence, in nickel and copper in thermomechanical treatment (TMT) reduction of internal friction background of the EI437A alloy depending upon degree of deformation was detected [12], whereby δ_0 value changed non-monotonously — it reduced at low TMT values and then achieved minimum and again increased.

Influence of hardening phase concentration in disperse-hardened materials on structure, microscopic limit of elasticity and internal friction. *NiCr–Al₂O₃ condensed materials.* In [3, 13] interconnection between the material structure and mechanical properties of NiCr–Al₂O₃ biphasic materials (microscopic limit of elasticity τ_s , plasticity ε at uniaxial tension, yield strength $\sigma_{0.2}$) is shown.

The materials were produced by deposition from vapor phase with application of electron beam evaporation of Ni20Cr ingots and pressed Al₂O₃ beads. The



condensates have columnar structure, Al_2O_3 particles are arranged in the Ni20Cr matrix rather uniformly and have practically spherical form. By means of Al_2O_3 content growth size d of the particles increased from 10 to 50 nm, size of the Ni20Cr matrix grain, determined by the width of columnar crystallites, reduced from 60 μm in the Ni20Cr matrix approximately to 2.0–2.5 μm at weight share of Al_2O_3 about 0.5–0.7 % [3, 13].

In this work interconnection between τ_s and δ and δ_0 characteristics was investigated. It was experimentally established that at content of the Al_2O_3 hardening phase from 0 to approximately 0.8 vol.% microscopic limit of elasticity τ_s and internal friction changed non-monotonously (Figures 2 and 3). It should be noted that behavior of τ_s , δ and δ_0 at low (0.1–0.2 vol.%) and higher (more than 0.2 vol.%) content of Al_2O_3 significantly differ. At 0.1–0.2 vol.% Al_2O_3 τ_s sharply reduces, achieving minimal value equal about 0.15 %. Assuming that at initial stage of microplastic deformation grain boundaries may act as sources of dislocations and taking into account the fact that by means of the hardening phase amount increase refining of the grain occurs (length of their boundaries increases), it is not excluded that reduction of τ_s is stipulated by growth of dislocation generation intensity. At volume share of Al_2O_3 approximately 0.15 % such size of grain D and the Al_2O_3 particles is achieved when the ratio

$$D = \Lambda; \quad \Lambda = \frac{2}{3} \frac{d}{f} \quad (2)$$

is observed. Here Λ is the mean free distance between the particles ($D = 8 \mu\text{m}$; $\Lambda = 7.5 \mu\text{m}$).

According to [13], in this area maximal value of plasticity was registered. At 0.15 vol.% Al_2O_3 dissipated energy achieves maximal value that matches presented in [3] minimal values of τ_s and $\sigma_{0.2}$ and maximal ε on curve of concentration dependence of $\sigma_{0.2}$ and ε . Effect of abnormal reduction of critical stress amplitude γ' , determined the beginning of microplastic flow, was established in investigation of influence of negligible share of zinc impurity on amplitude dependence of aluminium internal friction [14]. Reduction of the γ' value proves loss of the material strength that is confirmed by reduction of the microhardness level.

It should be noted that atoms of zinc in this case form coherently connected with the matrix segregations that generate new dislocations similar to carbide inclusions.

At 0.2–0.6 vol.% Al_2O_3 in structure of the condensates accumulations of dislocations were detected around Al_2O_3 particles that caused hardening of the material and increase of τ_s because of reduction of the number of sources able to generate dislocations at the preset stress. At content of Al_2O_3 approximately 0.8–1.0 vol.%, τ_s achieves maximal value (Figure 3), whereby dissipation of energy reduces and internal friction achieves minimal value.

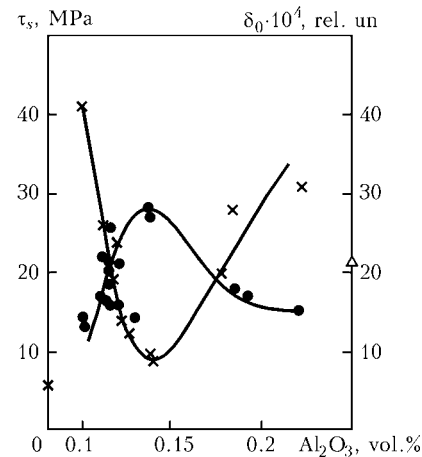


Figure 2. Influence of low content of second phase on τ_s and δ_0 in NiCr– Al_2O_3 condensed materials

At more than 0.8 % of volume share of Al_2O_3 , τ_s value reduces because of change of the mechanism that ensures beginning of microplastic deformation. In structure of the material matrix appear microtwins. As far as critical twinning stress is less than critical shear stress, process of propagation of dislocations proceeds at lower τ_s values, whereby it is accompanied by increase of the dissipated energy. Newly originated dislocations may be detained at boundaries of twins, forming accumulations with high local concentration of stresses. If sum of stresses (local + those applied to the specimen) achieve the level, necessary for origination of new dislocations, energy used for movement of originated dislocations significantly increases that is proved by increase of δ values by means of Al_2O_3 content growth.

Fe–Pb condensed materials. The Fe–Pb condensates with lead content up to 2 % have a bi-phased structure, consisting of a solid iron matrix and soft lead particles. Metallographic investigations showed that lead particles are uniformly distributed in iron matrix and have spherical shape and mean size of particles $d = 0.15 \mu\text{m}$.

It should be noted that character of τ_s , δ and δ_0 dependence upon lead content (Figure 4) is similar to that for NiCr– Al_2O_3 condensate (see Figure 3),

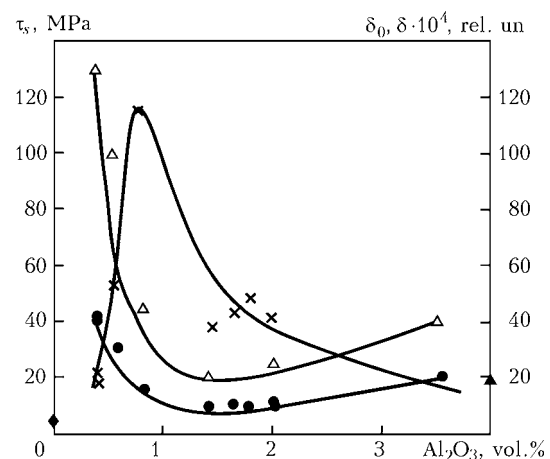


Figure 3. Influence of high content of second phase on τ_s , δ and δ_0 in NiCr– Al_2O_3 condensed materials

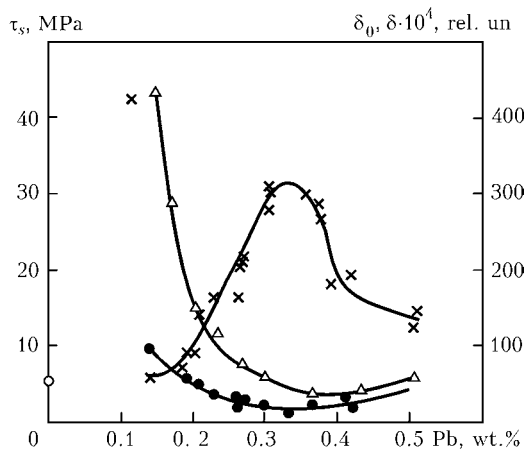


Figure 4. Dependence of τ_s , δ and δ_0 upon content of lead in Fe-Pb condensed materials

where NiCr matrix is hardened by solid Al_2O_3 particles.

Fe/Cu condensed microlayer materials. Fe/Cu microlayer condensates were produced by simultaneous electron beam evaporation in vacuum of iron and copper from two independent sources with subsequent condensation of vapors on a preliminarily heated up to 600 °C rotating substrate according to the method, described in [15]. Volume share of iron and copper constitutes approximately 50 %. Iron and copper layers had approximately equal thickness, which varied from 0.07 to 32.00 μm .

Investigated condensates represented a polycrystalline material with different structure of iron and copper [16]. Microlayers of iron have columnar structure, and thickness of grains is approximately equal to their thickness. Copper layers are characterized by equiaxial grains. Structure of interface between layers of different metals, which enter into composition of the condensate, is similar to the big-angle intergrain boundary in a polycrystal.

In [4] non-monotonous dependence of microscopic limit of elasticity τ_s upon thickness of the Fe/Cu layers is established, whereby internal friction also changes non-monotonously (Figure 5) and character of τ_s , δ and δ_0 dependence is stipulated by specificity of the condensate structure.

Value of τ_s within $h = 1.5\text{--}32.0\ \mu\text{m}$ stops to depend upon thickness of the Fe/Cu layers. Within this range of thicknesses the condensates are structurally stable. Length of the source, able to generate dislocations, calculated on basis of value of microscopic limit of elasticity (1), is 3–4 μm , which is commensurable with size of the condensate grains (subgrains). In this case interfaces between layers are not obstacles for movement and multiplication of dislocations, which causes both increase of the internal friction background δ_0 and growth of general energy dissipation δ .

At $h = 1.30\text{--}0.23\ \mu\text{m}$, when L_c practically coincides with the layer thickness, the material is hardened due to the fact that interfaces between the layers limit size of the grains and are an efficient barrier for movement of dislocations. Hardening is accompanied by

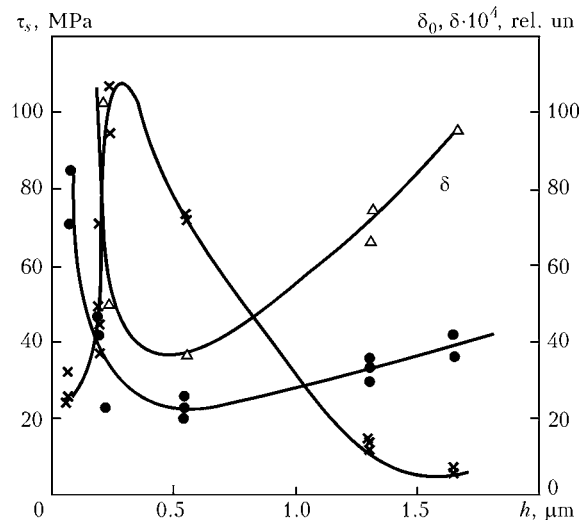


Figure 5. Dependence of τ_s , δ and δ_0 upon thickness h of layer in condensed microlayer Fe-Cu materials

increase of microscopic limit of elasticity, and τ_s achieves maximal value at $h = 0.23\ \mu\text{m}$. Registered reduction of internal friction occurs till microstructure of the condensates is characterized by structural stability. Minimal δ value corresponds to the τ_s maximum. It should be noted that at its minimum the internal friction background achieves the δ_0 value for pure iron, which proves that it is the carrying layer in the microlayer Fe/Cu condensate.

At $h \leq 0.19\ \mu\text{m}$ disintegration of the carrying layer (iron) occurs and the «bridges» are form between copper layers. This process is accompanied by loss of strength and respective drastic reduction of the τ_s value. Length of the dislocation source L_c significantly exceeds thickness of the layer h ($L_c = 0.76$ and $0.46\ \mu\text{m}$ respectively at $h = 0.07$ and $0.27\ \mu\text{m}$). This proves presence of the sources, able to generate dislocations at lower τ_s values, because in this case boundary between iron and copper layers is not obstacle for movement of newly originated dislocations and their penetration into adjacent layers.

It is not excluded that number of such dislocation sources may increase in case of disintegration of the microlayer condensate structure. More free movement of the dislocations causes significant increase of the internal friction background δ_0 and growth of energy consumption for microplastic strain δ_{pl} . General level of dissipated energy $\delta = \delta_0 + \delta_{pl}$ significantly increases.

Obtained experimental data match results of [3], in which significant increase of microhardness of the Fe/Cu condensate at $h = 0.20\ \mu\text{m}$ and sharp increase of strength at $h = 1\ \mu\text{m}$ with its maximal value being achieved at $h = 0.45\text{--}0.50\ \mu\text{m}$, were registered.

CONCLUSIONS

1. General dissipation of energy, as well as other mechanical properties of materials, are determined by their structure. The internal friction background δ_0 depends upon movement of dislocations, and microplastic strain losses δ_{pl} — mainly upon multiplication of dislocations.



2. It is shown that non-monotonous dependence of the energy dissipation characteristics at low-frequency oscillations upon parameters of the structure, obtained in materials with different prehistory, exists.

3. Size effect of mechanical properties (strength, plasticity) is established, provided linear dimensions of the structure elements (cells, grains, thickness of microlayers) and length of the dislocation free path are equal.

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INVESTIGATION OF ELECTRIC ARC UNDER CONDITIONS OF ITS INTERACTION WITH AIR-PULVERIZED COAL MIXTURE*

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Electric and energy parameters of three-phase system of AC arcs within wide range of technological conditions of air plasma conversion of the pulverized-coal fuel were investigated. Composition of the gas, formed in the process of conversion, was determined. It was revealed that to increase combustion heat of the converted gas (increase of hydrogen content and reduction of nitrogen content in mixture of the withdrawn from plasma reactor gases) it is necessary to perform air-vapor plasma conversion of the pulverized-coal fuel.

Keywords: *stabilization of burning, pulverized-coal fuel, air plasma conversion, plasmatron, arc, three-phase system of arcs, volt-ampere characteristic, converted gas*

Despite intensive development of alternative power engineering (solar, wind and other power stations, and production of biogas), fossil fuels will as ever remain main sources of energy. At present level of fuel consumption reserves of oil will be exhausted in 50 years, natural gas — in 60 years, and coal — in 220 years, which will cause rise in price of oil and gas. In comparison with coal, gaseous fuel has a number of advantages, for example, simplicity of transportation and regulated feeding in different technological processes. One should also take into account that application of coal in a number of processes is impossible because of presence of ash residue. In this connection efficient and economical gasification of coal is an actual task.

Gas may be used in many branches of industry, in particular, in metallurgy as a fuel and a reducer. Generating gas, produced in gasification of coal, may be used for stabilization of burning of the pulverized coal flame in furnaces of the power station and boiler house boilers. At present natural gas or fuel oil are used for this purpose, which, however, does not solve mentioned problem, because additional fuel has high cost and application of two different kinds of fuel complicates infrastructure of the enterprise.

As far as fuel oil is concerned, its application also causes worsening of environmental-economic parameters of a boiler: mechanical incomplete burning increases by 10–15 %, high-temperature corrosion of screen surfaces gets higher, emission of nitrogen and sulfur oxides increases by 30–50 % and forms divanadium pentoxide, and efficiency and reliability of the equipment reduces [1–3].

Lately the developments have been carried out, directed at application of plasma technologies for kindling of boilers and stabilization of the pulverized-coal flame burning. Application of plasma, in comparison with fuel oil technology, has a number of advantages: environmental-economic parameters of the boiler improve and reduces up to the permissible level (2–3 %) mechanical incomplete burning of the fuel. By means of the air mixture temperature growth, reaction capacity of the fuel and completeness of its combustion increase [1–6].

It was established in the course of previous investigations that for reliable ignition and stabilization of the air mixture burning power of the plasmatron is sufficient, which does not exceed 1 % of heat rate of the coal being ignited, and for anthracite it equals 2 %. Some authors consider that for stable operation of the system of plasma burning stabilization power of the plasmatron is sufficient, which constitutes 0.5 % of the torch power. By means of increase of the volatiles and concentration of coal in the air mixture specific energy consumption for plasma kindling reduces [1–7].

Optimal temperature of particles of the fuel, at which its reliable kindling and stabilization of the pulverized-coal flame burning are ensured, varies within 800–1200 K, whereby oxidizer excess factor should be 0.1–0.2. Efficient kindling of the fuel occurs at weight ratio of air and the fuel within 2.50–3.25, although in some cases this ratio was reduced down to 1 [1, 4, 7].

Depending upon ratio of air, oxygen, or water vapor, which are used for conversion of the pulverized-coal fuel, ratio of the conversion products may also change [8].

*Works were conducted within the bounds of Program «Energy saving».



As heat sources in plasma processes of kindling and stabilization of the fuel burning DC plasmotrons of indirect action are used [1–27], general shortcoming of which is low efficiency, increased heat loads on cathode and anode, and, because of this reason, short service life of the plasmotrons (50–100 h) [1, 2], their limited power, and need to stop the process for replacement of electrodes or plasmotrons. These plasmotrons have ascending volt-ampere characteristics (VAC) [5].

In practice water-cooled copper and graphite are used as materials for the plasmatron electrodes. In case of application as a plasma agent of air-pulverized coal mixture, erosion of copper electrode constitutes $(1.3\text{--}2.5) \cdot 10^{-6}$ g / C, and of the graphite one — $(4.0\text{--}5.6) \cdot 10^{-5}$ g / C. At arc current 200–1000 A arc voltage equals 280–400 V [5, 9–11].

Specific consumption of electric energy of plasmatron of indirect action for kindling and stabilization of the main flame achieves in the experiments 1 kW·h per 1 kg of coal, and ratio of masses of the pulverized-coal fuel and air in the air mixture, which is fed into the plasma jet, is maintained close to 1, whereby into the plasmatron 20 % of total mass of the fuel is fed, which is injected into the chamber [1, 2, 5].

At present plasma technologies are not widely used in power engineering. Main reasons of this are impossibility of ensuring continuity of the burning processes because of short service life of the plasmotrons, complexity of their installation in existing torches, slagging of working circuits of the torches, and increase of temperature of the components and units up to impermissible levels.

For introduction of plasma technologies into power engineering it is necessary to solve tasks of increasing service life of the plasmotrons and efficiency of mentioned process and to develop new methods for application of the plasmotrons. One of the ways of solution of the tasks, connected with application of plasma technology for kindling and stabilization of the pulverized-coal flame burning, is application of the multiarc plasma AC heaters with remote arcs [28–30], burning of which is directed at middle point in the plasma.

This approach is based on achievements of the E.O. Paton Electric Welding Institute in the field of development of three-phase AC plasma complexes, used as heat sources in processes of special electrometallurgy, and on results of investigations of peculiarities of the arc burning in case of application of natural gas or propane-butane as plasma gases. Application in metallurgy of multiarc plasma AC heaters with remote arcs, burning of which is directed at middle point in the plasma, is stipulated by their high efficiency and long service life and practically unlimited power. Operation of such heater envisages application of several arcs of big length, submerged into the flow being heated, whereby efficient mass exchange is ensured between high-temperature plasma jets and the flow, and heat, released in middle point of the plasma,

is efficiently used. All this predetermines high efficiency of the air-pulverized coal mixture heating. When burning of the arcs is directed at middle point of the plasma, the nozzle, designed for formation of the plasma jet of the air-pulverized coal mixture, is electrically neutral. Its heat loads are significantly lower than in plasmotrons of indirect action. Due to this service life of the nozzle is sufficiently high, power of the plasma heater is not limited by its service life and may increase due to increase of the number of arcs at value of current, at which high service life of the electrode is guaranteed. In the plasmatron design it is necessary to envisage build-up of the electrodes without de-energizing of power sources. It should be noted that alternating current sources are cheaper and simpler in operation than direct current sources.

In order to solve in complex issues, which prevent from using plasma technology, it is expedient to use the following scheme for construction of the complex of plasma kindling and stabilization of pulverized-coal fuel burning in furnaces of power boilers.

Near a power boiler or in the territory of a station a reactor is located for air, vapor, or air-vapor plasma conversion of the pulverized-coal fuel used at the enterprise. Plasma reactor is equipped with three- or multiphase system of AC plasma heaters, burning of arc of which is directed at middle point in the plasma. Plasma reactor, in addition to direct functions of fuel gasification, may catch and remove slag in order created due to conversion gas not contained particles, which may slag working circuits of the torch and the boiler. Gases, formed due to plasma conversion of the pulverized-coal fuel, are fed at preset temperature and pressure over pipeline into main torch and are combusted directly in the furnace space of the boiler in the area of outflow from the torch of main mass of the air-pulverized coal mixture. So, converted in plasma reactor gas fulfills function of kindling and stabilization of burning of main pulverized-coal mixture under most economical conditions, whereby design of main torch does not require for any adaptation. Heat conditions of operation of its components and units also changes insignificantly.

It follows from mentioned above that suggested scheme allows complete avoiding of shortcomings, peculiar to used plasma technologies and equipment.

Development of plasma technology for kindling of low-grade pulverized-coal fuel in furnaces of power boilers is impossible without prediction of electric and energy characteristics of the arc under conditions of interaction of plasma jets with air-pulverized coal mixture. It is necessary to know values of these parameters for ensuring stable burning of arc under conditions of kindling of low-grade pulverized coal fuel in furnaces of power boilers and determining of working range of parameters of the plasma arc heater–electric power source system.

Arc voltage in the process of kindling and stabilization of burning depends upon many factors —

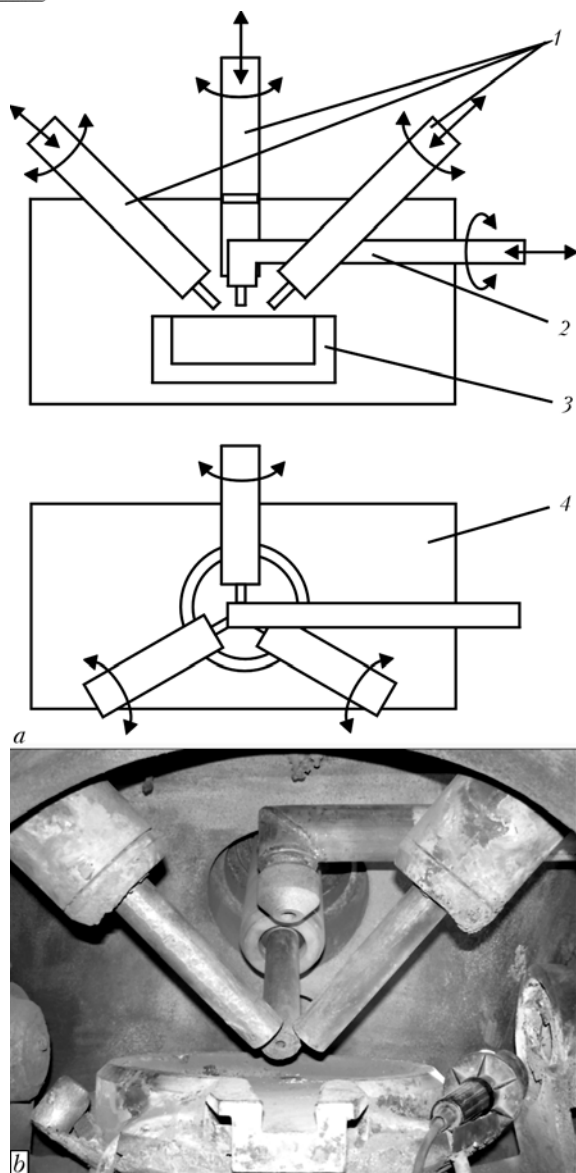


Figure 1. Scheme (a) and appearance (b) of experimental three-phase plasma reactor: 1 — plasma heaters; 2 — lance; 3 — slag catcher; 4 — chamber

composition and consumption of the air-pulverized coal mixture components, their humidity, arc length, etc. Depending upon range of the arc voltage changes at various periods of the process, requirements to stability of the arc burning, and its energy parameters open-circuit voltage of the power source and inclination of its external VAC are selected.

Purpose of this work is investigation and optimization of VAC and gradient of voltages of three-phase system of AC arcs within wide range of technological conditions, obtaining of empirical dependences for calculation of VAC, and determination of composition of gas formed in the process of plasma conversion of the pulverized-coal fuel.

Difference of the combustion process of each arc from the three-phase system of arcs from burning of a single arc consists in mutual influence of electromagnetic and thermal fields of arc columns, which determines certain peculiarity of their spatial arrange-

ment and movement and arrangement of active spots on the electrodes and areas being heated [28–30]. It should be noted that while for a single arc burning conditions in cold space are characteristic, for the three-phase system of arcs typical feature is burning in a hot space with partial compensation of energy consumption from the column of one arc by energy consumption from columns of adjacent arcs. Burning of the three-phase system of arcs in direction of middle point in the plasma also stipulates certain peculiarities of electrical conditions of these arcs. Presence of differences in conditions of burning of the three-phase system of arcs and a single arc caused need of comprehensive investigation of their generalized VAC within wide range of technological conditions.

For carrying out the experiments a three-phase plasma reactor was used for heating and conversion of the pulverized-coal fuel (Figure 1). Designs of the reactor and systems of its electric, water, gas and dust supply envisaged application of three plasma heaters 1 with hollow graphitized electrodes, uniformly installed over circumference and inclined to horizon (to surface of the slag catcher 3) in the air-tight chamber 4. Angle of inclination of plasma heaters was adjusted within 30–60°, and arc length within 0–450 mm. This ensured possibility of installation of plasma heaters in the position, in which arc burns in direction of the slag catcher bottom or middle point of the plasma.

For carrying out the experiments ranges of the arc current within 0.5–3.0 kA and open-circuit voltage of the three-phase power source within 220–575 V were sufficient. The power source represented a complex of the three-phase saturation choke and the isolation three-phase transformer. The power source had VAC with steepness of fall 0.30–0.45 V / A, which ensured stability of AC arc burning under conditions of significant variations of voltages of the arcs, stipulated by changes of the process technological parameters.

The water supply system was designed for cooling of units of plasma heaters, a slag catcher and a chamber, and the lance 2 (see Figure 1), designated for feeding air-pulverized coal mixture into the column and middle point of the three-phase system of arcs, its power being up to 2000 kW. Adjustment of the air-pulverized coal mixture composition and ratio of the components was performed by means of the air and dust supply systems, which ensured consumption of air and pulverized-coal fuel from 1 to 30 kg / h (each).

So, designed experimental three-phase plasma reactor ensured wide range of technological conditions for investigation of electrical and heat power parameters of three-phase system of arcs.

The ASH Donetsk anthracite was used as a fuel [31], main characteristics of which are presented below:

Dry mass, %	
Ash	18.0
General sulfur	1.9
Sulfate sulfur	0.1
Pyrite sulfur	1.1
Combustible mass, %	
Pyrite sulfur	1.4
Organic sulfur	0.8
Carbon	92.5
Hydrogen	1.8
Nitrogen	1.0

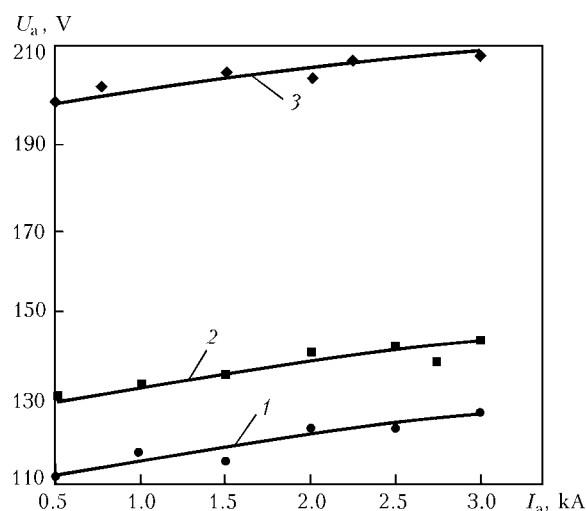


Figure 2. Family of arc VAC at $L_a = 10$ (1), 15 (2) and 30 (3) cm

Oxygen	2.5
Combustion heat of combustible mass, kJ / kg	
In bomb	33820
The lowest	33150
Yield of volatiles, %	4
Lowest heat of fuel combustion	25120

The reactor is equipped with instruments for measuring current and voltage of arcs of each plasma-arc heater and air and dust consumption in the lance. In addition, into the branch pipe, designed for withdrawal of gases that formed in the process of heating and conversion of air and pulverized coal, a discharge pipe was installed for taking samples of gases, analysis of composition of which was made using the «Gazokhrom 3101» chromatograph.

Results of investigations of main arc discharge electrical parameters under conditions of interaction of plasma jets of the three-phase system of arcs with air-pulverized coal mixture depending upon arc length L_a , flow rate G of the air-pulverized coal mixture and ratio of its components, and pressure P_{ch} of gases in the reactor chamber are shown in Figures 2–6. In the experiments, in addition to stipulated cases, flow rate of air G_{air} was 1 kg / h, and coal dust G_c — 0.32 kg / h.

As one may see from the Figures, character of the VAC curves of the three-phase system is faintly ascending, their slope (steepness) varies within $(3.6\text{--}6.0) \cdot 10^{-3}$ V / A, and gradient of the arc voltage

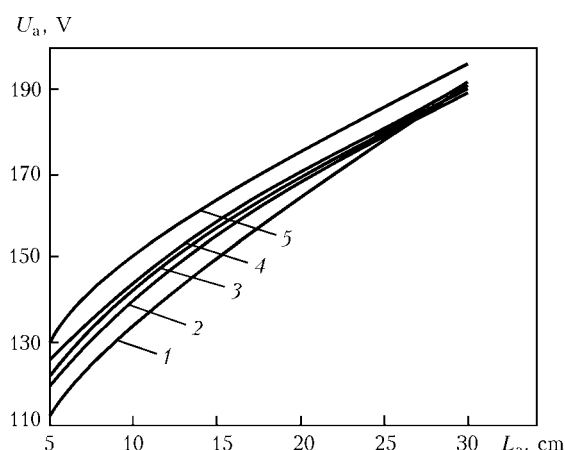


Figure 3. Dependence of arc voltage U_a upon its length L_a at $I_a = 1.0$ (1), 1.5 (2), 2.0 (3), 2.5 (4) and 3.0 (5) kA

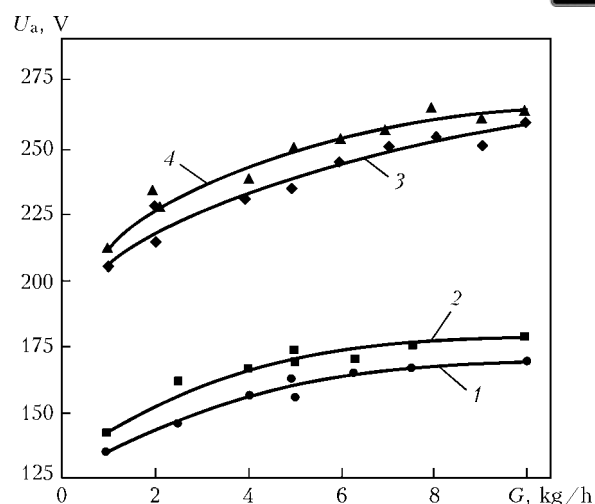


Figure 4. Dependence of arc voltage U_a upon consumption G of air-pulverized coal mixture: 1 — $I_a = 1$ kA, $L_a = 15$ cm; 2 — $I_a = 3$ kA, $L_a = 15$ cm; 3 — $I_a = 1$ kA, $L_a = 30$ cm; 4 — $I_a = 3$ kA, $L_a = 30$ cm

ΔU_a — within 2.7–10.0 V / cm. Inclination of VAC and gradient of the arc voltage depend upon flow rate and composition of the air-pulverized coal mixture and pressure of gases in the reactor chamber.

Maximal VAC steepness was noted at a comparatively small length of the arcs, and by means of their length increase it reduced, because in this case reduced stabilization influence of plasma mixtures on arc columns, due to which increased diameter of their current conducting area. As a result, mutual stabilization of burning of each arc gets more noticeable due to compensation of their heat losses by heat losses from columns of adjacent arcs, which causes reduction of electrical resistance of the arc columns. Trend towards growth of VAC and arc voltage gradient was noted in case of increase of the plasma air-pulverized coal mixture consumption and pressure in the reactor chamber. This happens because of increase of energy consumption of the arc columns for heating and ionization of the mixture, and because of increase of energy consumption from arc columns by means of density increase of the gases, in environment of which

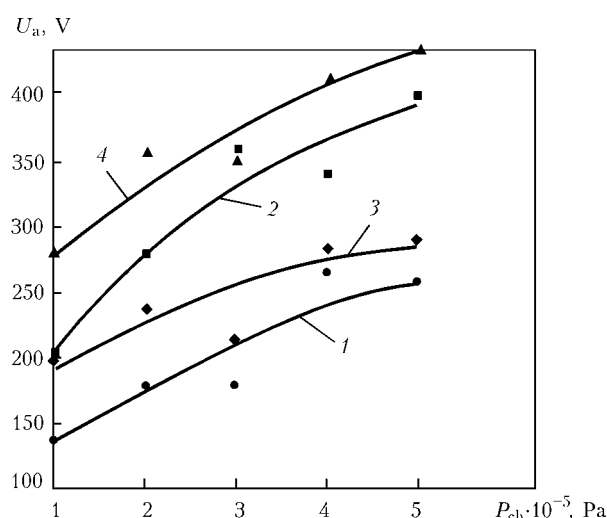


Figure 5. Dependence of arc voltage U_a upon pressure P_{ch} of gases in chamber: 1 — $I_a = 1$ kA, $L_a = 15$ cm; 2 — $I_a = 1$ kA, $L_a = 30$ cm; 3 — $I_a = 3$ kA, $L_a = 15$ cm; 4 — $I_a = 3$ kA, $L_a = 30$ cm

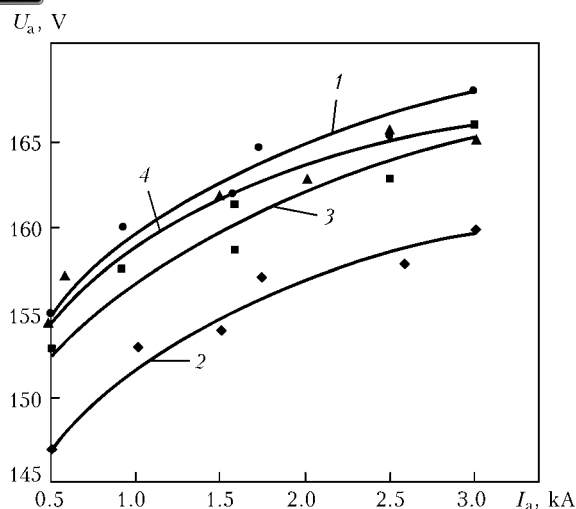


Figure 6. Family of arc VAC at different consumptions of air-pulverized coal mixture components ($L_a = 15$ cm): 1 — $G_{air} = 5.8$ kg / h, $G_c = 1.8$ kg / h; 2 — $G_{air} = 5.8$ kg / h, $G_c = 3.7$ kg / h; 3 — $G_{air} = 11.6$ kg / h, $G_c = 2.1$ kg / h; 4 — $G_{air} = 11.4$ kg / h, $G_c = 5.5$ kg / h

burn the arcs. By means of reduction of the air and pulverized-coal fuel mass consumption, gradient of arc voltage also reduces. Evidently, getting of the current-conducting pulverized-coal fuel into the arc column enables reduction of its electrical resistance.

Values of arc voltage and VAC shape of the arcs in case of their burning in direction of the current-conducting surface and middle point of the plasma, technological parameters being varied, practically do not differ.

As a whole it should be noted that over the whole range of variations of current, arc length, composition and ratio of components of air-pulverized coal mixture, as well as pressure of gases in the reactor chamber, the three-phase system of arcs burned stably and fluctuations of voltage and strength of current did not exceed 15 % of their mean value.

Analysis and generalization of experimental electrical parameters of the three-phase system of arcs allowed obtaining empirical dependence for calculation of their VAC within wide range of variation of current and length of arcs, as well as gas pressure in the reactor chamber:

$$U_a = 2.2(2.3L_a I_a^{1/L_a^{1.05}} + 10)(1 \cdot 10^{-5} P_{ch})^n G^m \text{ [V]}, \quad (1)$$

where n , m are the indices of the degree, which are selected respectively within 0.35–0.43 and 0.05–0.10 ranges (smaller values of indices of the degree correspond to smaller ratios of mass consumptions of air and pulverized coal).

Table 1. Chemical composition of gas (vol.%) produced due to plasma air conversion of pulverized-coal fuel (anthracite)

Experiment No.	Consumption, kg / h		Air fuel: ratio	$\hat{I}_2$	$\hat{N} \hat{I}$	$\hat{N} \hat{I}_2$	N_2
	Air	Fuel					
1	7.5	1.1	6.80	2	8	21	69
2	7.5	1.6	4.70	3	22	5	70
3	15.0	2.5	6.00	3	13	14	70
4	15.0	3.9	3.85	8	25	2	65

Comparison of VAC, obtained according to formula (1), and data of the experiments showed that calculated values of voltages match well the experimental ones.

One of the most important energy parameters of the three-phase system of arcs is its power. Within investigated range of values of current (0.5–2.5 kA) it varied from 470 to 1250 kW. This power is quite sufficient for kindling and stabilization of the torch fuel burning of the biggest acting power stations and furnaces of power boilers [1–7]. Calculation confirmed that within this range of power it is easy to ensure within 1 h heating up to 1000 °C and conversion of 1300–3500 kg of air-pulverized coal mixture.

Carried out investigations showed that specific erosion of a hollow graphitized electrode of the plasma-arc heater at $I_a = 0.5$ –3.0 kA does not exceed $5.0 \cdot 10^{-5}$ g / C even at unfavorable ratio of mass consumptions of air and fuel. Evidently, by means of reduction of this ratio one should expect reduction of erosion of the electrodes.

On basis of results of the investigations one may calculate main initial electrical and energy parameters of the plasma heating complex and power source of the three-phase reactor for heating and conversion of the air-pulverized coal mixture: $I_a = 1$ –3 kA; phase open-circuit voltage of the three-phase power supply source — 550 V; steepness of the power supply source external VAC — not less than 0.5 V / A.

One of the most important indices of efficiency of plasma air conversion of the pulverized-coal fuel is composition of the produced gas. Investigations of its composition (Table 1), depending upon consumption and ratio of air and pulverized coal (anthracite), were carried out. Produced gas was taken for analysis at outlet from the chamber before ventilation system. As it was mentioned earlier, concentration of H_2 , CO and CO_2 gases was measured by the «Gazokhrom 3101» chromatograph. Evidently, the remained gas mixture component is N_2 , which constitutes basis of air.

Stoichiometrically for the pulverized-coal fuel of this composition ratio of air and the fuel should equal 5 in order to convert the whole carbon into monoxide. Greater ratio may cause increase of CO_2 content, which is proved experimentally. In the experiments optimal ratio was about 4. This may be explained by increase of unaccounted air together with the pulverized coal, deposition of the latter in some areas of the dust duct, and incomplete burning of carbon in the chamber, whereby, naturally, efficiency of the gasification process reduced. Mentioned shortcomings may be avoided by improvement of the dust supply system of the reactor and application of the chamber, lined with refractory materials, which will cause increase of temperature of gases in the chamber and completeness of the fuel combustion.

In the produced gas rather small amount of hydrogen is present that is explained by insignificant content of volatiles in anthracite. It is evident that in case of using coal with a higher content of volatiles volume share of H_2 will increase. Increased content of hydrogen in experiment No. 4 (Table 1) may be explained by presence of moisture in the air and the



dust. It follows from this that for increasing content of hydrogen in mixture of gases, withdrawn from the reactor, conversion of the pulverized-coal fuel by air enriched with water vapor, should be performed.

In Table 2 calculated composition and calorific value of generating gas in case of conversion of coal containing 75 vol.% C and minimal amount of volatiles is presented. One may see from the Table that the highest specific calorific value of the generating gas is observed in oxygen conversion, and maximal calorific value of gas, produced from 1 kg of solid fuel, in vapor conversion.

CONCLUSIONS

1. VAC curves of the three-phase plasma heating complex with hollow graphitized electrodes under conditions of interaction of plasma jets with the air-pulverized coal mixture increase insignificantly. Their inclination varies within $(3.6-6.0) \cdot 10^{-3}$ V/A, and gradient of the arc voltage under these conditions — within 2.7–10.0 V/cm.

2. Inclination of the VAC curves and gradient of the arc voltage depend upon consumption and composition of the air-pulverized coal mixture and pressure of gases in the reactor chamber.

3. Optimal mass ratio of the air and the pulverized-coal fuel mixture in air plasma conversion is 4–5, whereby mixture of CO + H₂ gases is formed with the content not less than 25 vol.%.

4. For increasing the converted gas combustion heat (increase of hydrogen content and reduction of content of nitrogen and mixture of gases withdrawn from the plasma reactor) it is necessary to perform air-vapor or air-oxygen plasma conversion of the pulverized-coal fuel.

Table 2. Calculated composition (vol.%) of gas in different kinds of pulverized-coal fuel conversion

Oxidizer	H ₂	CO	N ₂	Calorific value, kJ	
				Per 1 kg of anthracite	Per 1 m ³ of gas
Air (3.5 m ³)	0	33	67	17560	4180
Oxygen (0.7 m ³)	0	100	0	17560	12540
Steam (1.4 m ³)	50	50	0	32600	11700

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INDUCTION MELTING IN SECTIONAL MOULD, ITS POSSIBILITIES AND PROSPECTS OF APPLICATION FOR PRODUCTION OF CAST ITEMS

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Peculiarities of the melting process in a sectional mould with application of an induction source of heating, possible methods of molten metal accumulation in a sectional mould and its casting are considered. Ways of intensification of the molten metal melting and overheating and possible assortment of cast items are shown.

Keywords: inductor, electromagnetic field, molten metal, metal pool, sectional mould, casting mould, casting

Induction heating is rather widely used in different fields of engineering, including metallurgy, for heat treatment, growing of microcrystals, and melting of metals and alloys due to complex of the following unique technological properties, which characterize induction source of heating:

- absence of negative influence on composition and properties of the materials being heated;
- intensive mixing of molten metal during melting allows ensuring equalizing of temperature and chemical composition within the pool volume;
- absence of local overheating of the metal enables insignificant melting loss of the alloying elements;
- absence of rigid connection between fed electric power and rate of melting makes it possible to keep metal melt in molten state for a long time and control solidification of the metal;
- creation of any special conditions for operation are not necessary, that's why the technological process may proceed in gaseous atmosphere of any composition and pressure, including vacuum;

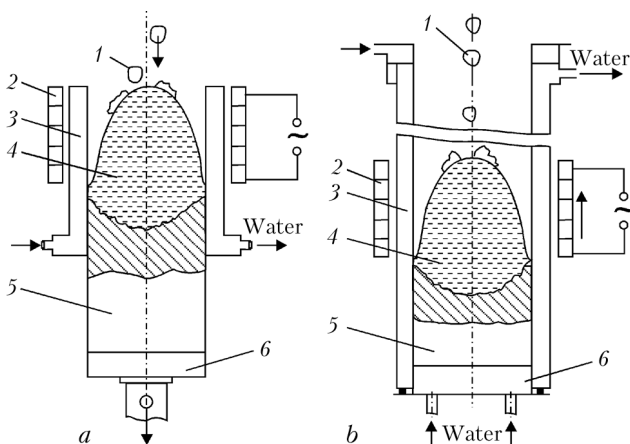


Figure 1. Principle schemes of IRSM: *a* — melting of ingot by its pulling from mould; *b* — melting of ingot by its building-up in mould; 1 — charge; 2 — inductor; 3 — sectional mould; 4 — metal pool; 5 — ingot; 6 — bottom plate

- high reliability of the heating source operation is ensured due to absence in it of consumable elements, for example, electrodes.

However, in the existing melting furnaces mentioned advantages of the induction heating source are not implemented to a full degree, because during melting the molten metal contacts with a refractory wall of the crucible and is contaminated by the interaction reaction products (non-metal and gas impurities). Intensive mixing of the melt in this case accelerates the process.

This, certainly, limits application of the induction furnaces for melting and refining of highly reactionary metals and alloys, for example, titanium, REM, etc.

A progressive method for melting of ingots with application of the induction heating sources is induction remelting in a sectional mould (IRSM), developed in the E.O. Paton Electric Welding Institute of the NAS of Ukraine [1–3]. This process (Figure 1) consists in the fact that in a sectional mould, the wall of which is «transparent» for electromagnetic field, high-frequency electromagnetic field is created, in which metal pool is induced by melting of a lumpy charge or a monolithic inoculation. Further building of an ingot is carried out by melting of the lumpy charge, which is fed into the metal pool batchwise, whereby volume of the metal pool in the process of melting remains unchangeable, and the ingot is pulled out from the mould or built up in the mould, for which purpose the inductor and, therefore, the metal pool are moved from downwards upwards. So, the process is clearly oriented at melting of the ingots, external configuration of which depends upon shape of the mould (cylindrical, rectangular, etc.).

Characteristic feature for IRSM is that height of the inductor does not exceed height of internal diameter of the mould ($h_{ind} \leq D_m$, where h_{ind} is the inductor height, D_m is the mould diameter). In this case optimal volume of the metal pool $V_{m.p}$ is, in its turn, connected with frequency of current in the inductor, diameter of the mould and constitutes 0.6 of volume of the mould, limited by height of the inductor, i.e. [1]



$$V_{m.p} \approx 0.15\pi D_m^2 h_{ind} \approx 0.15D_m^2 h_{ind}.$$

It follows from presented relation that volume of molten metal, accumulated in the mould, depends upon its diameter and height of the inductor. Smaller volume of metal pool, in comparison with volume of the mould limited by height of the inductor, is explained by forcing out of the metal melt from wall of the mould by electromagnetic field created by current of the inductor (Figure 2).

This volume of metal pool is quite sufficient for forming an ingot with external surface of good quality and crystalline structure (Figure 3). However, IRSM allows producing ingots of mainly round, rectangular, or square section.

Another interesting direction, in which induction heating is used without contact of molten metal with refractory materials, is melting in a cold crucible [4–7], which differs from conventional crucible induction melting by conditions of energy transfer from the inductor to a melt, because between the inductor and the metal melt a wall of the cold crucible is located, which distorts electromagnetic field created by current in the inductor.

A continuous metal wall of a conventional cooled crucible is the screen for high-frequency electromagnetic field created by the inductor, because eddy currents induced in wall of the crucible form on its external surface a closed electric circuit. Wall of the cold crucible, made from separate electrically isolated cooled sections, represents secondary winding of a transformer. Due to this fact occurs transfer of the electromagnetic field energy from the inductor to the melt in a cold crucible [4, 6, 7].

Despite the fact that a portion of the electromagnetic field energy is lost in cooled wall of the crucible, rather high value of the cylindrical inductor power factor (in comparison with the end one) ensures release of energy in the charge sufficient for its melting and subsequent overheating of the metal melt. Uniform side heating and intensive mixing of the melt in electromagnetic field allow getting pool with homogeneous chemical composition and temperature.

In case of induction melting in the cold crucible geometric parameters of the latter are close to those of the crucibles, made from refractory materials, used in conventional induction furnaces, i.e. height of the crucible and the inductor exceeds diameter of the crucible 2–3 times. That's why for induction melting in the cold crucible big depth of the metal pool is characteristic. Over height of the metal pool, located in the cold crucible, one may single out three characteristic zones (Figure 4) [8]. Upper one *I* is the zone of complete forcing out of the melt from the cold crucible wall; middle one *II* is the zone of partial (slot) forcing out of the melt from the crucible wall; and the lower one *III* is the zone of full contact of the melt with the cold crucible wall.

In this direction owing to efforts of many scientific-research and design institutes of the former Soviet Union (VNIETO, VNIITVCh, NIAT, IMET,

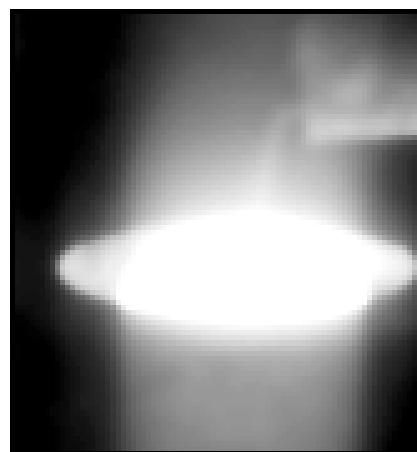


Figure 2. Convex meniscus of metal pool in IRSM of titanium ($D_m = 70$ mm; $f = 66$ kHz)

VIAM, the A.I. Lylka SPA «Saturn», etc.) a number of induction foundry furnaces with cold crucibles from 5 to 100 kg (titanium) capacity were developed and mastered under conditions of semi-commercial operation. On these units energy conditions of melting mainly titanium and its alloys were developed, energy, heat, and technical-economic parameters of operation of the units were determined, main parameters and requirements for power sources, selection of current frequency, etc. were established.

Foundry units of this kind (with the cold crucible) represent a vacuum or a gas-filled chamber, inside of which are installed water-cooled sectional crucible with inductor and a platform or a carousel for placement of casting moulds.

Casting of metal in such installations is performed with application of the bottom pouring (Figure 5, *a*) or through the cooled pouring lip by rotation of the crucible around axis of rotation (Figure 5, *b*).

Majority of foundry furnaces with cold crucible (UPKhT-1, UPhkT-2, UPhkT-3) are made according to the principle of the metal pouring by means of inclination (rotation) of the melting module around

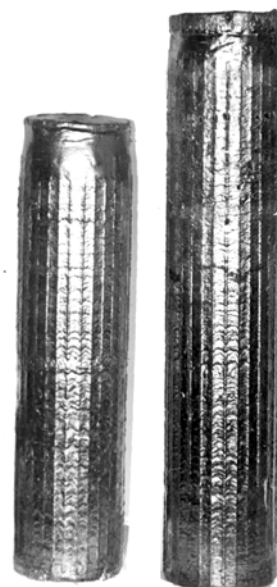


Figure 3. Appearance of titanium ingots of 100 mm diameter

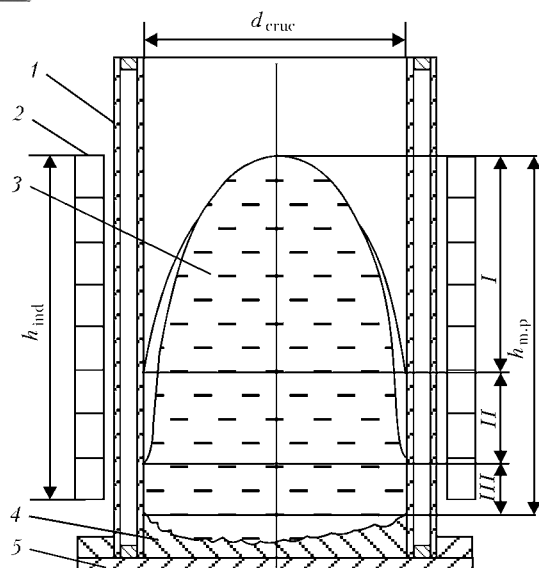


Figure 4. Characteristic zones of metal pool in melting in cold crucible: I — full forcing out of melt from crucible wall; II — slot forcing out of melt; III — full contact of melt with crucible wall; 1 — cold crucible; 2 — inductor; 3 — metal pool; 4 — skull; 5 — bottom

axis of rotation [7]. Melting modules of the UPKhT-1 and UPKhT-2 installations are placed into horizontal cylindrical melting chambers with two end doors, which ensure their reliable air-tightness in melting and convenient servicing of the melting module during preparatory period (Figure 6). These are installations of periodic action, i.e. after each cycle, which includes loading of the charge into the crucible, sealing of the melting chamber, melting and pouring of the metal, depressurization of the melting chamber is performed for removal of casting moulds and preparation of the installation for the next melt.

(Titanium) capacity of the UPKhT-2 cold crucible is 100 kg.

The UPKhT-2 casting module is equipped with resistance elements, which allows warming up casting moulds up to 1100 °C and producing high-precision thin-wall casting, for example, from high-strength structural and high-temperature nickel-base alloys for aviation engine building.

Universality of UPKhT-2 is not limited by production of shaped casting from various metals and alloys by application of different methods of pouring (stationary moulds or chill moulds, centrifugal casting or casting into a hot mould). The installation may be used for melting of alloys of rare metals, hardeners, brazing alloys, etc. in the form of cast billets of small weight from primary charge materials or wastes of own production used as the charge.

When changing the alloy or the metal to be remelted, it is necessary to change residual skull, formed in the course of previous melts, by means of disconnection of the bottoms from the crucible and knocking-out of the skull in pouring position of the crucible, i.e. inclined (horizontal) position of the crucible.

Two OPCh-500-2.4 machine frequency converters, which work separately or in parallel on one load of total power 1000 kW, are used as power sources of the UPKhT-2 installation.

On basis of the UPKhT-2 installation the UPKhT-2M furnace was designed. According to statement of authors of the development, this is a small-size melting unit of laboratory-commercial designation with improved arrangement of the components, designed for melting of chromium. The melting module with cold crucible of about 25 kg capacity is powered from three 160 kW vacuum-tube frequency converters.

In addition to melting of alloys from primary charge, one may remelt in UPKhT wastes of any kinds (including chips, sponge, and crop ends) at different percent ratio thereof in the charge (up to 100 %) with their introduction into crucible by free charging, i.e. openly before a melt.

Despite significant energy inputs, characteristic of the furnaces of this type (with the cold crucible), which in some cases exceeds 5 kW·h / kg, technological and metallurgical possibilities allow their using for melting of alloys from primary charge and remelting of different wastes and produce final product in a single remelt without subsequent conversions.

It should be noted that use of the melting units as induction foundry furnaces with cold crucible for production of a metal melt of preset chemical compo-

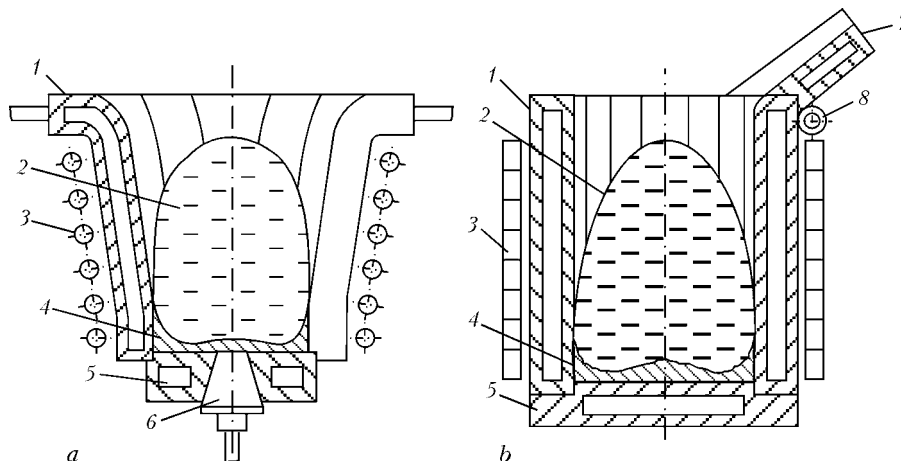


Figure 5. Schemes of melting units of casting installations with cold crucible: a — bottom pouring of metal; b — pouring of metal by rotation of cold crucible; 1 — cold crucible; 2 — metal pool; 3 — inductor; 4 — skull; 5 — bottom plate; 6 — device for controlling pouring of metal (plug); 7 — pouring lip; 8 — axle of rotation

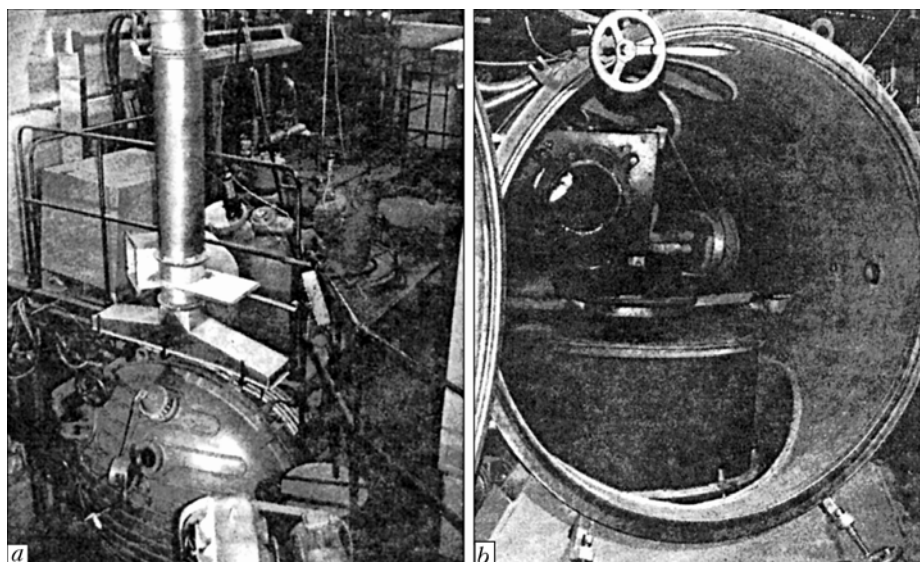


Figure 6. UPKhT-2 casting induction installation with cold crucible [7]: *a* — general view; *b* — internal space of melting chamber

sition allows, in contrast to conventional induction crucible furnaces, melting alloys on basis of titanium, chromium and other high-reaction metals, because metal melt does not enter into chemical interaction with cold wall of the crucible.

Design of cold crucibles of different modifications and capacity has common principle, i.e. side cylindrical wall is assembled from water-cooled copper sections, electrically insulated from each other over adjacent planes. Such design excludes induction of ring currents over external surface of the crucible sleeve and at the same time allows inducing in each section of ring currents in the inductor zone. In this case the melting module, including the inductor and the cylindrical or conical sleeve of the crucible, forms a unified energy system, by means of which electromagnetic field, created by the inductor current, is transferred to the charge, whereby internal wall of the crucible acts as inductor for the charge.

Main shortcoming of cast units with the cold crucible consists in increased specific energy inputs, which achieve in certain cases 7–8 kW·h / kg of poured metal. That's why when designing such melting units it is necessary first of all to solve issues of energy economy and adaptability for manufacture of the design as a whole, whereby issue of melting of the metal is not principal in this case, it is rather well studied and energy inputs for melting of certain metals and alloys are quite acceptable. Final technical-economic result is determined by casting of the metal, because in the best case not more than 60 % of metal gets into the casting mould [5, 9–11]. In the course of casting almost half of the molten metal freezes on cooled elements of the melting module (wall, bottom, pouring lip of the cold crucible). In addition, it is necessary to solve a number of specific issues, such as cleaning of the crucible pouring lip and the hole in the crucible bottom, prevention of formation of bridges, stabilization and minimization of skull in bottom area of the crucible, etc. All this proves complexity of designing highly efficient and economical melting units of this type. Despite the fact that many issues of

induction melting in cold crucibles are well studied both in theoretical and design respect, the latter did not find wide practical application for manufacturing of castings in industry.

Certain steps in investigation and expansion of technical possibilities of the induction melting processes in the cold crucible and sectional mould were made at the E.O. Paton Electric Welding Institute, NAS of Ukraine. Three versions of production of hollow billets on laboratory equipment and semi-commercial OP-117 installation were tested. General technological principle of the tested methods is production of a hollow billet of the bushing type, i.e. having ratio $h_b / D_b \leq 1$ (where h_b and D_b are respectively the height and diameter of the billet). In tested methods wall of a sectional mould acted as a forming element of side surface of the billet, and internal cavity was formed by means of a rod from a refractory material. Role of the latter in all cases played graphite.

A scheme for production of hollow metal billets by melting of a measured consumable billet, installed before melting into a sectional mould on upper end of the rod, is presented in Figure 7. At the beginning of melting a consumable measured billet is introduced by means of a mobile bottom plate from below into the inductor, and its upper part is submelted, whereby molten metal is retained by electromagnetic field on the lower non-molten part (a pedestal).

Then a consumable billet with a rod is introduced by means of the bottom plate into the inductor. Further melting of the consumable billet occurs, and formed molten metal is poured on the bottom plate forming a hollow billet.

A hollow billet may be also produced by introducing a rod from a refractory material from above into the metal pool (Figure 8). For the metal pool formation the following steps may be used:

- melting of a measured consumable billet with a known in advance chemical composition;
- melting of a lumpy charge of the same grade or of the charge, consisting of materials with different chemical composition.

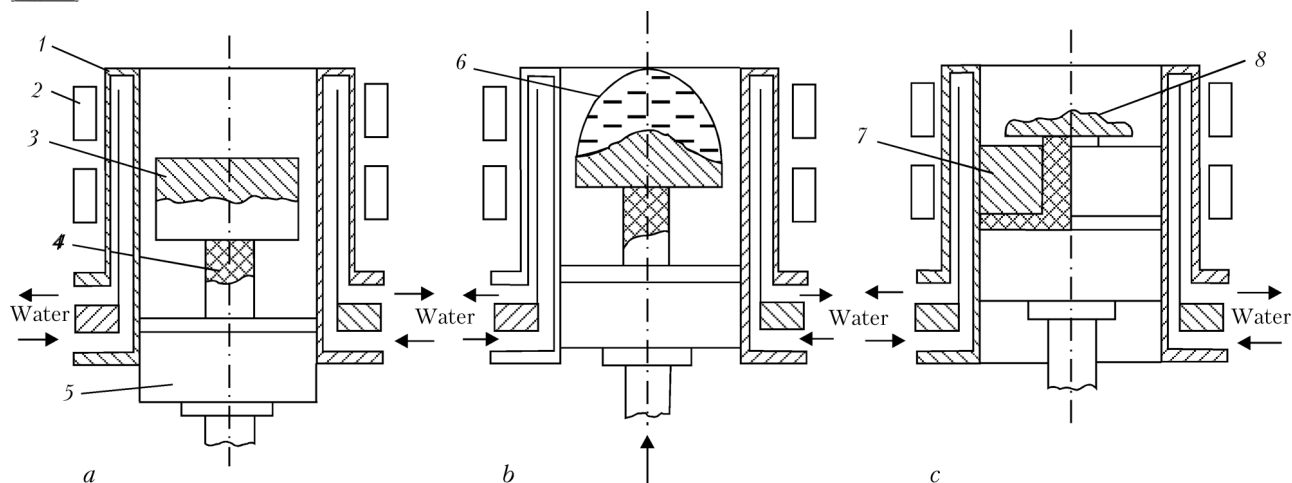


Figure 7. Operation-by-operation scheme of producing hollow ingots-billets of bushing type by melting of measured billet on rod: *a* — location of rod and billet in sectional mould; *b* — melting of billet; *c* — solidification of metal in mould; 1 — sectional mould; 2 — inductor; 3 — measured consumable billet; 4 — graphite rod; 5 — bottom plate; 6 — metal pool; 7 — hollow casting; 8 — residual skull

Appearance of the produced in this way hollow billet is shown in Figure 9.

Scheme of melting of an annular billet from the OT4-2 titanium alloy on the OP117 semi-commercial installation is presented in Figure 10. Essence of this method consists in the fact that a graphite rod is fixed on a bottom plate in the sectional mould. Ring space between the rod and the mould wall is filled by the lumpy charge. Waste annular elements of the casting fitting-out were used in this work (Figure 11). Then the charge was melted by means of electromagnetic field, created by current of the inductor (Figure 10, *b*). Partially forced out from the mould wall molten metal completely covered the rod. After switching off

of the electromagnetic field the molten metal settled down and filled ring space between the rod and the mould wall (Figure 10, *c*).

Appearance of the produced annular billet is shown in Figure 12.

When hollow billets were produced by described methods, their mass corresponded, approximately, to mass of the homogeneous metal pool, i.e. in one melt could be produced one cast billet, which is not always economically justified.

In [12] rather promising method for producing billets is described, which is based on principle of the induction metal melting and retaining of the produced melt on the support by electromagnetic forces. Essence

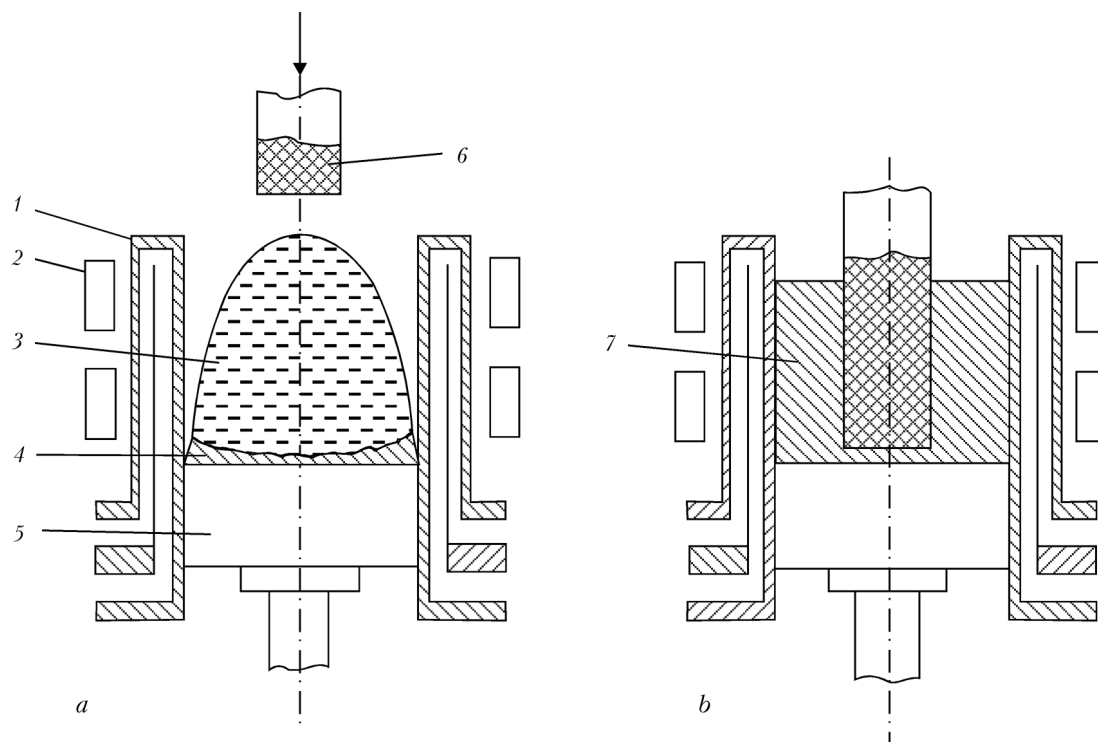


Figure 8. Operation-by-operation scheme of producing hollow ingots-billets of bushing type by formation of cavity by movable rod: *a* — forming of metal pool; *b* — solidification of metal with introduced rod; 1 — sectional mould; 2 — inductor; 3 — metal pool; 4 — bottom skull; 5 — bottom plate; 6 — movable rod; 7 — hollow billet



Figure 9. Appearance of hollow billet of 70 mm diameter with rod

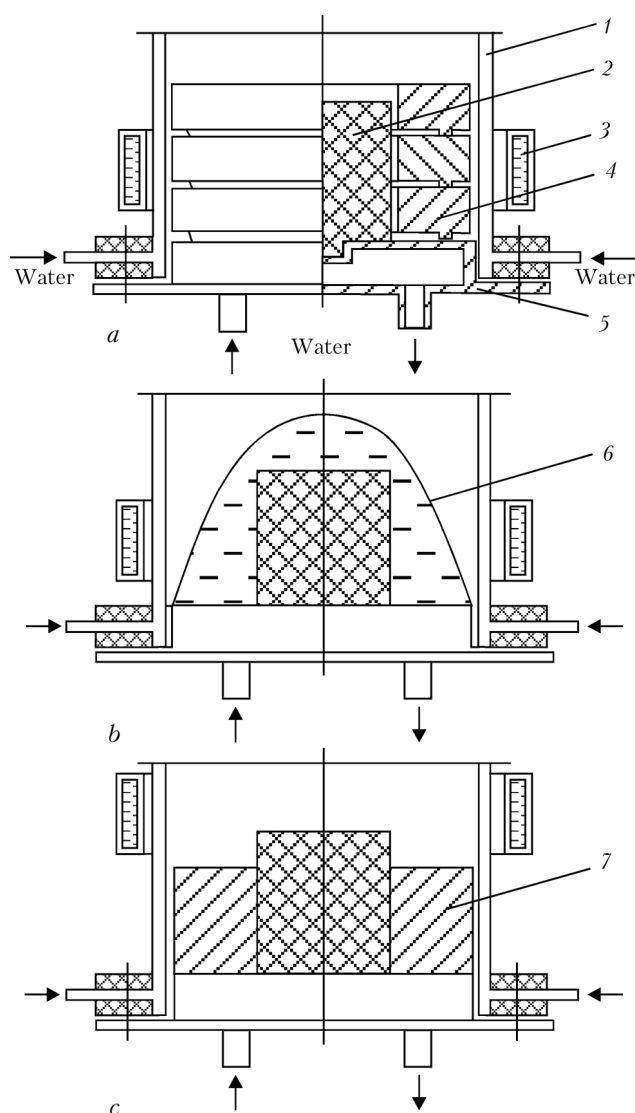


Figure 10. Operation-by-operation scheme of melting hollow ingots-billets of washer type on OP117 semi-commercial installation: *a* — location of charge and rod in sectional mould; *b* — melting of charge; *c* — solidification of metal; 1 — mould; 2 — graphite rod; 3 — inductor; 4 — charge; 5 — bottom plate; 6 — molten metal (pool); 7 — cast billet

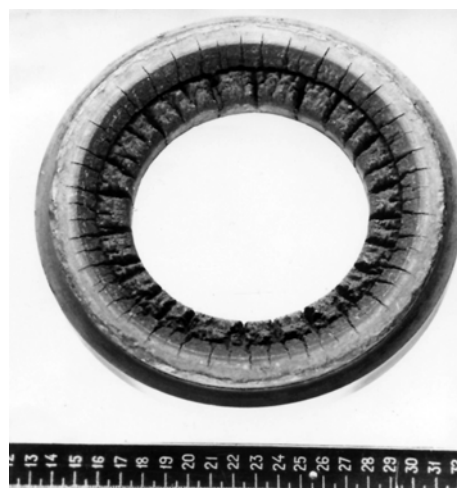


Figure 11. Appearance of used elements of casting fitting-out from OT4-2 alloy

of this method consists in the fact that a consumable measured billet with a preset chemical composition is placed in the inductor by means of a cooled bottom plate made in the shape of a ring (Figure 13). A mould is coaxially installed under the inductor. Melting of the billet is performed using the inductor. First upper part of the billet is melted, and molten metal is retained by electromagnetic forces on lower non-molten part of the billet, which functions as a support.

By means of the metal pool temperature increase thickness of the support portion reduces and, finally, an instant occurs, when it melts through and molten metal is poured into the mould.

In regard to its technical possibilities, including purity of the melted metal, induction melting on a support is identical to that of the metal in a suspended state. At the same time presence of a support under the melt allows retaining in electromagnetic field rather big mass of the metal.

By developing this idea for induction melting of metal one may use a melting assembly, consisting of a sectional mould of small height and an inductor (Figure 14). This will allow excluding non-controlled spills of the metal from periphery of the metal pool. Application of carousels for location and movement of consumable billets and casting moulds allows performing without depressurization of the melting



Figure 12. Casting-billet of 220 mm diameter with graphite rod produced in IRSM installation

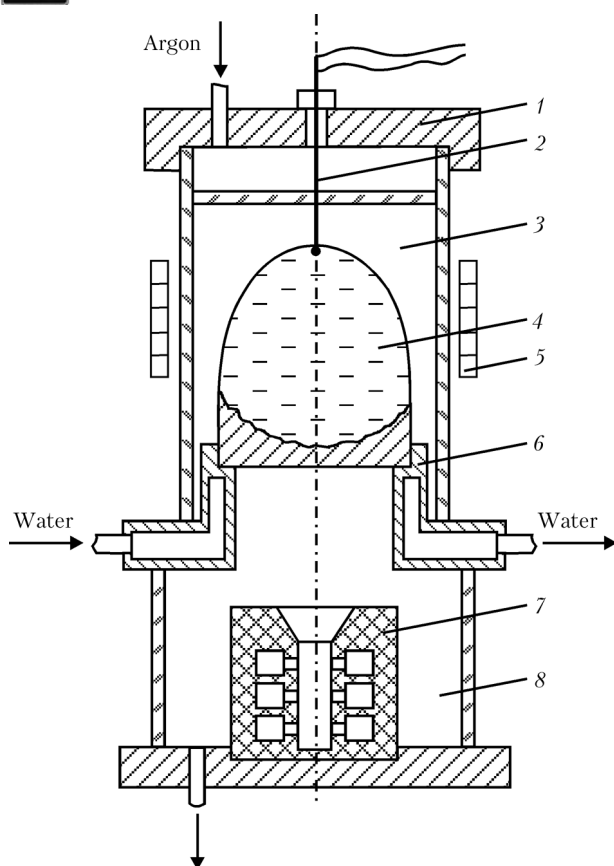


Figure 13. Scheme of installation for induction melting of metals on support and casting of items: 1 — cover; 2 — thermocouple; 3 — melting chamber; 4 — metal pool; 5 — inductor; 6 — cooled ring (bottom plate); 7 — casting mould; 8 — chamber for moulds

chamber several melts and castings, which enables increase of productivity of the installation and its technical-economic parameters.

When designing casting installations of this type it is necessary to take into account that overheating of the metal in presence of a sectional mould or a cold crucible is insignificant. As a rule it does not exceed 50–60 °C, which is for casting processes, especially for production of small castings, apparently insufficient, whereby increase of power, supplied to the inductor, does not cause automatic increase of the metal pool temperature, because in this case increases force action on the metal melt and the latter is forced more out from the mould wall [1]. So, heat-receiving surface of the metal pool moves into the electromagnetic field area having lower intensity. So, increase of heat release in the pool does not occur, and its temperature does not increase.

One of the ways to increase the metal pool temperature, necessary for producing castings of good quality, may be equipment of the melting-casting units with additional heat sources, for example, plasma or electron beam ones. Intensive mixing of a metal pool, characteristic of induction heating, will enable fast heat transfer and equalization of temperature within the pool volume.

Additional plasma heating is successfully used for intensification of melting in induction furnaces with ceramic crucibles [13]. In addition, complementary

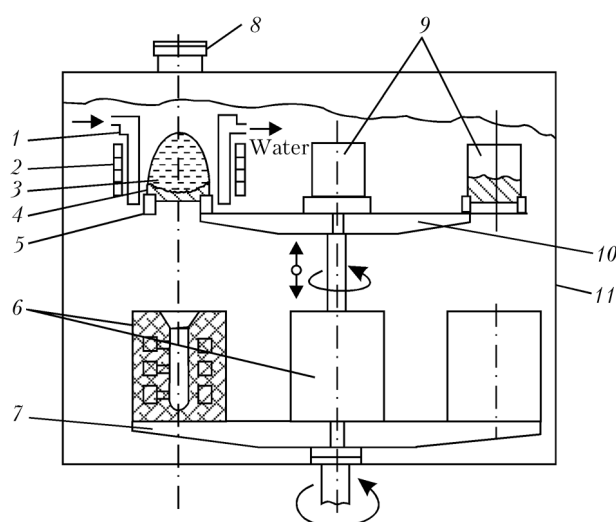


Figure 14. Scheme of induction installation for production of cast items: 1 — sectional mould; 2 — inductor; 3 — metal pool; 4 — skull; 5 — bottom plate-ring; 6 — casting moulds; 7 — carousel for casting moulds; 8 — inspection system; 9 — measured consumable billets; 10 — carousel for consumable billets; 11 — air-tight (melting) chamber

arc heating is used for remelting of metals with high melting point in induction installations with a sectional mould [14].

So, when designing induction melting-casting installations with a melting module on basis of a sectional mould, one has to take into account many technical parameters and technological possibilities of the process, including possibilities of equipment of the installation with additional high-concentration heat sources, for example, an arc plasmatron or an electron beam gun.

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ON DISSOLUTION OF TiN SOLID PARTICLES IN MOLTEN TITANIUM DURING MELTING

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Interaction of molten titanium with crucible from titanium nitride at temperatures 2000 and 2100 °C was investigated. It is shown that at these temperatures occurs intensive interaction of the crucible material with the molten pool. The results obtained give basis to assume possibility of dissolution of the titanium nitride solid particles in the molten titanium.

Keywords: titanium, titanium nitride, dissolution, induction heating

Quality of service properties of items from titanium and titanium-base alloys depend to a great degree upon presence in it of non-metal inclusions, in particular nitrides of different chemical composition, formed during production and remelting of titanium sponge and subsequent production of titanium alloys. Due to this in the metal in addition to titanium nitride may form so called «enriched by nitrogen inclusions», which represent, as a rule, compound of TiN_x type, surrounded by layers of α - and β -Ti with increased content of nitrogen. In the literature they are called «solid» alpha-inclusions or «low density inclusions» that are characterized by hardness, which is several times higher than that of metal titanium, and because of this reason they are places of origination and activation of cracks at various kinds of loads, that causes reduction of fatigue strength of manufactured from them parts of structures [1]. Presence of such inclusions in titanium represents significantly greater problem in comparison with general content of dissolved in the metal nitrogen. This problem is aggravated even more by complexity of detection of such inclusions even by methods of ultrasonic and X-ray analyses.

Existence of mentioned problem contributed to appearance of a number of works, directed at investigation of possibility of removing and preventing formation of the «low density» nitride inclusions as early as at the stage of molten titanium. Very first investigations showed existence of the «phenomenon» of solid TiN dissolution (melting point is 3290 °C) in molten alloy on basis of the Ti-6242 titanium at temperature 1760–2000 °C. The investigations were carried out in electron beam and plasma-arc remelting. However, the most promising technology for removal of the TiN particles is considered electron beam remelting [2–4]. In these works additions of solid TiN of stoichiometric composition or «synthetic» TiN with different contents of nitrogen were introduced into the molten titanium pool. In [5] mechanism of nitride

dissolution in titanium is proposed. Reaction of interaction is described by equation



Theoretical substantiation of possibility of progress of this process is presented in [6]. At the same time mechanism of solid TiN fracture is not quite clear. It was assumed that it could occur because of both dissolution of titanium nitrides and their deposition in the molten pool [7]. In addition, influence of oxygen, contained in pressed «synthetic» TiN, was not taken into account. In majority of cases during investigation of dissolution of solid TiN particles, the TiN pellet was introduced into volume of molten titanium, whereby volume of molten titanium significantly exceeded volume of solid TiN pellets. Such ratio may cause quick distribution of the TiN particles over the whole volume of the pool, which makes difficult precise determination of the mechanism of solid TiN fracture in molten titanium. In this connection more interesting looks experiment «on the contrary», when volume of molten titanium is concluded in the volume of solid TiN. In our opinion such approach will make it possible to determine by certain signs (state of the interphase boundary, presence of solid inclusions in molten titanium, their form and chemical composition) more accurately mechanism of dissolution and fracture of solid TiN.

We tried to directly investigate process of TiN interaction with molten titanium and possibility of dissolution of solid particles in the molten metal. We investigated interaction of molten titanium with the crucible, made from titanium nitride, in induction melting. The crucible with titanium was placed into the reaction chamber and left within the whole experiment till full cooling (Figure 1). The experiments were carried out in argon. Content of nitrogen in the crucible material was 22 wt.%, and in iodide titanium — 0.008 wt.%. Temperature was controlled by the HOT SHOT color pyrometer. It was 2000 and 2100 °C, and duration of the experiments was 7 min

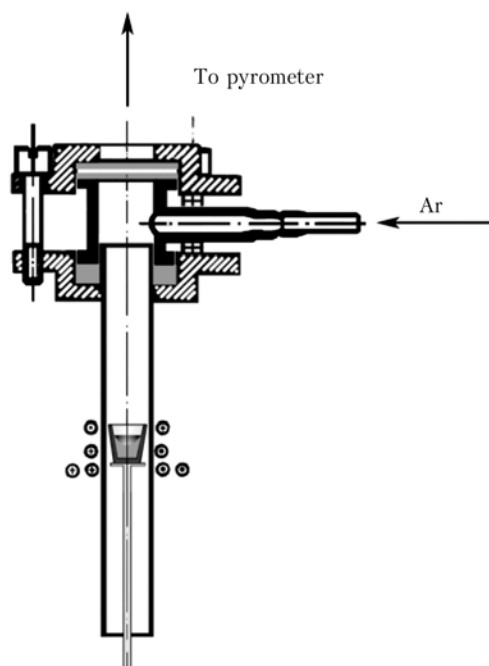


Figure 1. Scheme of reaction chamber

from the time of achievement by molten titanium of the preset temperature.

In Figure 2 general view of the crucible after melting at temperature 2000 °C is presented. Just in 30–40 s after beginning of heating titanium was in molten state and intensively mixed with inductive flows. In 2 min on external side of the crucible wall the molten metal was detected, which was the result of formation in the crucible wall of a «gully». After increase of temperature of the experiment up to 2100 °C (Figure 3) this «gully» appeared approximately in 1 min 40 s, which proves increase of intensity of interaction between molten titanium and material of the crucible walls.

During cooling of the metal up to its solidification its surface remained quite. After solidification it had shape of a concave meniscus, which proved good wetting of the crucible walls. Cracks in bottom part of the crucible (Figure 2) and on internal surface of side walls (Figures 3 and 4) formed in the process of cooling.



Figure 2. General view of crucible after melting

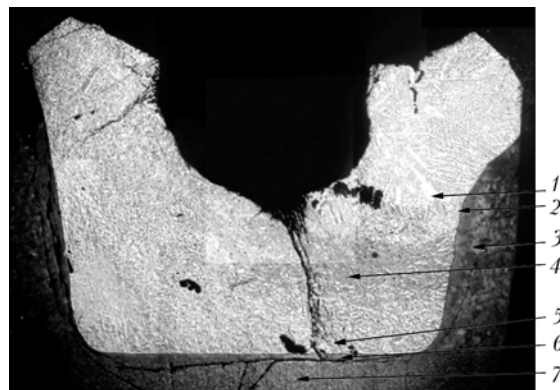


Figure 3. Cross section of specimen at temperature of experiment 2100 °C: 1 — 2.9; 2 — 15.0; 3 — 18.0; 4 — 4.6; 5 — 13.0; 6 — 13.4; 7 — 19.9 wt.% N ($\times 16$)

Contents of nitrogen and oxygen, dissolved in titanium, were determined on the LECO gas analyzers TN-114 and RO-316. Samples for the analysis were taken from central zone of the specimens. General content of nitrogen in titanium was 2.08 wt.% at temperature 2000 °C, 5.2 wt.% at 2100 °C, and content of oxygen equaled respectively 0.16 and 0.24 wt.%. Amount of oxygen in the crucible itself changed as follows: at temperature 2100 °C it constituted in the crucible walls 0.64 wt.%, and in bottom part — 0.75 wt.%; at 2100 °C it equaled respectively 0.58 and 0.75 wt.%.

In Figure 3 cross section of the specimen is presented after melting at temperature 2100 °C. Surface of this specimen was not subjected to etching. Dark area in central upper part of the Figure shows place where the metal was taken for gas analysis. Cracks in side walls and bottom part of the crucible formed during cooling, which is proved by a rather loud crackling sound. In right upper part of the Figure is the area, where molten metal has «eaten through» the crucible side wall up to the external edge. Structure over the whole section of the specimen is inhomogeneous (for example, in upper left and right parts of the Figure).

In this connection rather interesting is investigation of nitrogen distribution over the whole section of the specimen and determination of its content in separate phases, which was carried out on the Camebax SX-50 X-ray microanalyzer (Figures 3 and 4).

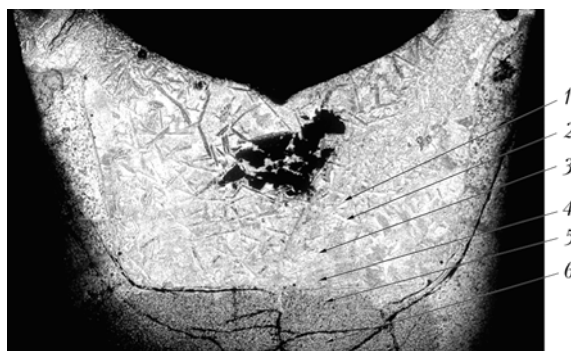


Figure 4. Cross section of specimen at temperature of experiment 2000 °C: 1 — 0.50; 2 — 0.65; 3 — 1.58; 4 — 2.16; 5 — 13.30; 6 — 19.53 wt.% N ($\times 16$)

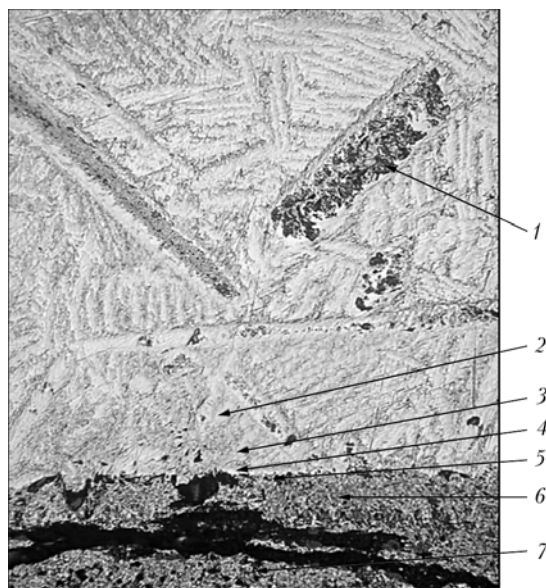


Figure 5. Bottom part of specimen at temperature of experiment 2000 °C: 1 — 13.10; 2 — 1.58; 3 — 2.16; 4 — 11.16; 5 — 9.82; 6 — 13.30; 7 — 19.53 wt.% N ($\times 160$)

It should be noted that in periphery zones of the crucible bottom and walls content of nitrogen (approximately 20 wt.%) is close to its content in initial state. By means of approaching interface of the solid body–molten metal phases it diminishes down to 9.82 at 2000 °C and 10 wt.% at 2100 °C, which is explained for induction melting by different magnetic permeability of titanium and titanium nitride, which is significantly higher in titanium, and because of this reason temperature at the interface of the solid body–molten metal phases is much lower than on the crucible surface.

Of a certain interest is investigation of interface of the Ti–TiN phases. The interface is clearly pronounced over the whole length (Figures 4 and 5) and has the form of thin light-lemon strips on side of the crucible and light strips on side of titanium. Nitrogen in this area is distributed as follows, wt.%: crucible — 9.82 and 10.24 at temperature of the experiments 2000 and 2100 °C respectively. Content of nitrogen in the light-lemon strip on side of the crucible is 11.16 at 2000 °C and 13.4 wt.% at 2100 °C; content of nitrogen in the light strip on side of titanium at the same temperatures was respectively 2.16 and 3.80 wt.% (Figures 5 and 6). By means of getting away from

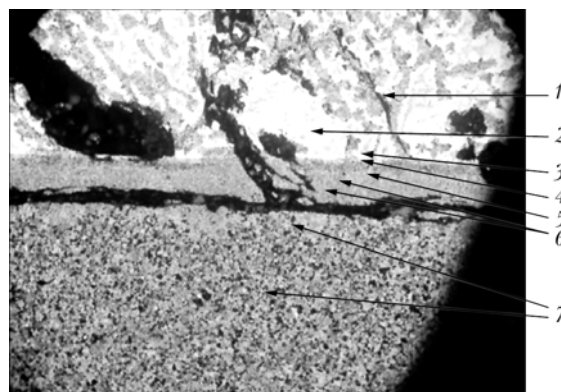


Figure 6. Bottom part of specimen at temperature of experiment 2000 °C: 1 — 13.00; 2 — 2.90; 3 — 3.79; 4 — 14.20; 5 — 10.20; 6 — 13.40; 7 — 19.90 wt.% N ($\times 160$)

the interface to center of the specimens content of nitrogen in titanium increased from 0.5 to 4.0 wt.% by means of the temperature increase.

At temperature of the experiment 2000 °C orderly arranged accumulations of secondary nitrides were detected over the whole section of the specimen, where content of nitrogen achieved up to 13 wt.%. After increase of temperature of the experiment up to 2100 °C content of nitrogen in secondary nitrides was the same, but they were arranged chaotically which is, evidently, connected with increased convective flows.

The results obtained give basis to assume possibility of dissolution of solid TiN particles in molten titanium, whereby rate of dissolution increases by means of temperature increase.

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DIFFUSION COMPLEX COATINGS WITH ADDITION OF TITANIUM ON HARD VK8 AND T15K6 ALLOYS

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Results of investigation of phase and chemical compositions, structure and microhardness of the carbide-base complex coatings with addition of chromium, nitrogen and oxygen on hard VK8 and T15K6 alloys are presented. It is shown that depending upon the method of diffusion saturation multilayer carbide (Cr_{23}C_6 , TiC) or carbooxide (TiC, Ti_xO) coatings are formed on surface of hard alloys. It is established that stability of multifaceted non-resharpenable plates with complex coatings increases in cutting in comparison with the serial ones 1.4–1.5 times.

Keywords: diffusion coatings, carbide, nitride, oxide, titanium, hard alloys, stability in cutting

At present hard wear-resistant coatings on basis of carbides, nitrides and metal oxides are widely used for increasing stability of multifaceted non-resharpenable hard alloy plates (MNHP) [1–3].

Advantages of MNHP with the coatings are evident [1–4], in particular, a longer period of operation: MNHP with the coatings may serve 10–20 times longer than without the coating, depending upon type of the latter, initial, alloy and field of application. In addition, MNHP with the coatings demonstrate the best results at higher speeds of cutting than the initial ones. Optimal speed of cutting increases up to 90 %. At the same time quality of the surface being processed is improved [1, 3, 4].

At present for increasing stability of hard alloys both single- and multilayer coatings on basis of titanium carbide and nitride and aluminium oxide, produced by CVD method, are widely used [3, 4]. Less distributed are coatings from hafnium nitrides and carbides, chromium and zirconium carbides, titanium boride, etc. [3–5]. At different versions of arrangement of TiC, TiN and Al_2O_3 layers in a multilayer coating maximum implementation of such properties as heat resistance, low coefficient of friction, wear resistance, etc. is possible [4, 5]. Selection for MNHP of the best type of a coating (a multi- or a single-layer one) is determined by the hard alloy grade and conditions of operation.

New generation of the protection coatings should have high (not less than $HV50$ 40 GPa) hardness and heat resistance [1, 6, 7]. Amount of materials with such properties is limited. So, amorphous boron carbide with hardness up to $HV50$ 50 GPa is rather brittle and is intensively oxidized even at 460 °C [6, 8].

Cubic boron nitride and hemicrystalline diamond films are also characterized by high hardness, but they are metastable and are oxidized rather easily.

Advisability of application of multilayer coatings with participation of nanostructural and amorphous

materials requires for confirmation by results of the carried out tests. In addition, effect of high hardness of multilayer coatings disappears rather quickly, which is most probably connected with diffusion dispersion of separate layers [7, 9]. Influence of multilayer coatings on the phenomenon of relaxation under conditions of increased temperatures is not quite clear.

Possibility of producing various coating–base compositions with necessary structure, composition and properties is, evidently, limited first of all by technological peculiarities of the coating application methods.

Of a certain interest as a material for protection coatings are oxides and oxycarbonitrides of transition metals of IV–VI groups of the periodic table [10, 11]. Till recent time certification of such compounds with participation of oxygen was incomplete that did not allow drawing conclusion about nature of protection properties of the coating. Oxygen is one of the most distributed interstitial impurities, which enables formation of superstoichiometric compounds and exerts significant influence on properties of the interstitial phases [10, 12]. Presence of oxygen increases hardness of the $\text{TiC}_{0.98}$ carbide by $HV50$ 5 GPa, and $\text{TiC}_{0.8}$ — by $HV50$ 11 GPa [11, 12]. At the same time hardness of the $\text{Ti}(\text{C}_{0.86}\text{O}_{0.14})$ oxycarbide, produced by ion-plasma method, is $HV50$ 28.2 GPa, which is by $HV50$ 10.8 GPa less than hardness of the $\text{TiC}_{0.85}$ carbide, produced by chemical deposition from gaseous phase [7]. So, hardness and other properties of coatings on basis of carbides, oxycarbides and oxycarbonitrides of transition metals depend to a great degree upon technology of their production.

Expediency of forming on the MNHP surface of titanium-base oxide layers is shown in [10]. During cutting on surface of the TiC coating thin layers of oxides with protection properties are formed, which isolate a tool from the alloy being processed. Good adhesion of oxide layers of titanium with the TiC coating is enabled by formation of a transition zone on basis of titanium oxycarbides.

**Table 1.** Phase composition and properties of coatings with addition of titanium on VK8 and T15K6 alloys

Kind of treatment	Grade of alloy	Duration of saturation, h, at $T = 1050\text{ }^{\circ}\text{C}$	Phase composition	Lattice spacing, nm	Thickness of coating, μm	Microhardness of coating HV_{50} , GPa
Titanizing	VK8	2.0	TiC	0.4323	6.0	37.5
	T15K6	2.5	TiC	0.4326	5.5	37.0
Titanizing in combination with oxidizing	VK8	2.0	TiC	0.4318	5.0	39.0
			Ti_3O , Me_2TiO	—	1.5	—
Titanizing in combination with chromization	VK8	2.5	TiC	0.4288	4.0	37.0
	T15K6	2.5	Cr_{23}C_6	—	2.5	—
			TiC	0.4315	4.0	36.5
			Cr_{23}C_6	—	1.5	—

So, purpose of this work is development of new methods for application of complex coatings with addition of titanium, chromium, oxygen and nitrogen on surface of hard VK8 and T15K6 alloys, designated for cutting tools, and investigation of composition, structure, properties and characteristics of MNHP with the coatings.

Protection coatings were applied on surface of the VK8 and T15K6 alloys at temperature $1050\text{ }^{\circ}\text{C}$ with soaking for 2–4 h in a closed reaction space at reduced pressure with application of powders of titanium and chromium, carbon tetrachloride and carbon-containing additive as initial reagents [2].

Oxidizing of titanized alloys is performed by a single introduction of air into the reaction space at the saturation temperature. Chromization was performed in a single technological cycle. At first stage chromization at temperature $1050\text{ }^{\circ}\text{C}$ within 1–2 h, at second stage titanizing at temperature $1050\text{ }^{\circ}\text{C}$ within 1–3 h were performed. Depending upon technological peculiarities, suggested processes of the diffusion saturation received the following names: titanizing by the known method [2, 13], chromization in combination with titanizing, and titanizing in combination with oxidizing. Metallographic, durometric, X-ray diffraction and X-ray microspectral investigations were carried out using known methods of physical materials science.

Results of investigation of phase composition, structure and some properties of the coatings on hard VK8 and T15K6 alloys are presented in Table 1.

Analysis of the data obtained showed that phase composition of the coatings is to a significant degree determined by the method of chemical-heat treatment and to smaller degree — by the hard alloy composi-

tion. Layer-by-layer X-ray structural analysis showed that in titanizing and titanizing in combination with oxidizing TiC titanium carbide layer is directly adjacent to the base, while in chromization in combination with titanizing — Cr_{23}C_6 layer.

It should be noted that difference of lattice spacing of the TiC carbide layer, produced by different methods, is, probably, stipulated by presence in the TiC carbide of different weight shares of carbon, oxygen and nitrogen. Presence of the $\text{Co}_3\text{W}_3\text{C}$ carbide-base layer was not detected, which is connected with participation in formation of carbide layers not just of the base carbon, but also of the saturation medium carbon.

Macroanalysis of hard alloys after chemical-heat treatment showed that titanizing and chromization in combination with titanizing are in majority of cases accompanied by formation of coatings of the dark-grey color. Titanized-oxidized layers are usually light-dim with a small metallic luster. Laminations and cracks in coatings on hard alloys were not detected.

Investigated in the work coatings are detected on cross microsections in the form of a light zone with well pronounced coating–hard alloy interface, whereby in the coatings, subjected to titanizing in combination with oxidizing, a layer on basis of titanium oxides turns out to be somewhat lighter than the TiC carbide layer. Microstructure of fracture surfaces of coatings on VK8 alloy is presented in Figure 1. For the produced coatings brittle inter- and transcrystalline fracture are characteristic. So, the TiC carbide-base layer in case of titanizing [2], titanizing in combination with oxidizing and chromization with titanizing consists of equiaxial grains, size of which does not exceed $0.2\text{--}0.8\text{ }\mu\text{m}$. Shape and size of the

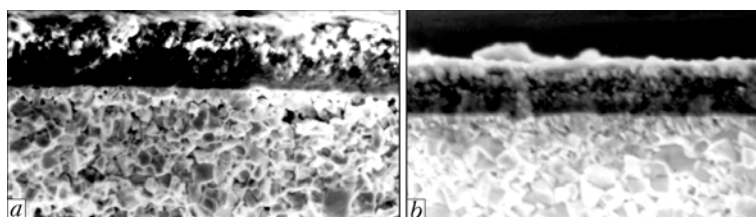


Figure 1. Microstructure of fracture surfaces of coatings on VK8 alloy (SEM): a — titanizing in combination with oxidizing ($\times 3000$); b — chromization in combination with titanizing ($\times 5400$)

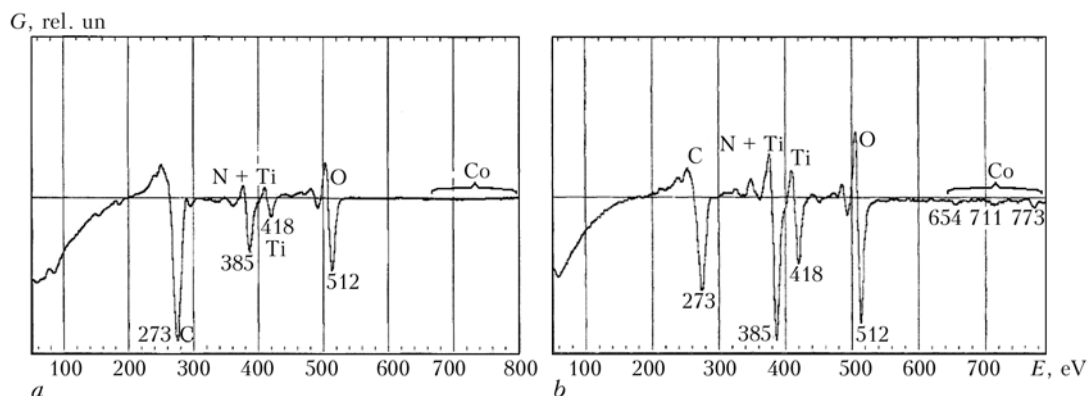


Figure 2. Auger-spectra of titanized-oxidized coating on surface of VK8 alloy (a) and at distance 180 nm from its surface (b): *G* — intensity; *E* — energy of electrons

grains in regard to the thickness of separate layers practically do not change.

Comparative local mass-spectral analysis of titanized and oxidized-titanized layers on the EKHO-4M installation with a laser probe for determining content in certain zones of the coatings of oxygen and carbon showed that content of oxygen in central zones of the TiC-base coating after titanizing in combination with oxidizing was somewhat higher than after titanizing, whereby content of oxygen in the zone of oxides sharply reduces in transition through the oxide-carbide boundary.

Using method of Auger-spectroscopy, character of oxygen, nitrogen, carbon and titanium content change in thin zones on external side of the coatings was determined (Figure 2). Content of carbon is maximal on the surface and monotonously diminishes over depth of the layer, whereby increase of weight share of oxygen and nitrogen occurs. At distance 180 nm from the surface traces of cobalt appear on spectral curves of the coating. So, one may consider that de-

termined by radiographic method phase $\text{Me}_2(\text{Ti}_3\text{O}, \text{Ti}_4\text{O})$ is oxycarbonitride of complex composition.

As showed analysis of results of the investigations, microhardness of coatings on hard alloys depends to a great degree upon the method of saturation. Maximal microhardness was achieved in the TiC coating on the VK8 alloy after titanizing in combination with oxidizing. Data of [2, 7, 8, 14] prove significant differences in microhardness of coatings of the same type, which is connected both with peculiarities of technological methods of saturation and structure of the coatings, as well as methodological difficulties of the investigations. Presented in the work values of microhardness may reflect chemical dissimilarity of composition of the coating over its thickness, which is confirmed by change of electron structure of the coatings on basis of the transition metal carbides [14].

In order to determine influence of protection coatings on serviceability of MNHP the authors carried out industrial cutting tests of different grades of the steels processed (Table 2).

Table 2. Influence of protection coatings on MNHP stability in sharpening

Grade of hard alloy	Material processed	Kind of processing	Cutting conditions			Coefficient of cutting speed increase
			<i>v</i> , m / s	<i>S</i> , mm / rev	<i>t</i> , mm	
Ø15K6	Steel 20	Titanizing	5.3	0.128	1.0	1.4
		Titanizing in combination with oxidizing				2.0
		Chromization in combination with titanizing				1.9
	Steel ShKh15	Titanizing	1.3	0.200	0.5	2.0
		Titanizing in combination with oxidizing				5.0
		Chromization in combination with titanizing				4.5
VK8	Steel 40Kh	Titanizing	2.2	0.200	0.2	2.5
		Chromization in combination with titanizing				2.6
	Steel U8A	Titanizing	3.3	0.430	1.0	2.7
		Titanizing in combination with oxidizing				13.5
		Chromization in combination with titanizing				8.2

Note. *v* — cutting speed; *S* — feeding speed; *t* — depth of cutting.



Cutting properties of plates with the coatings were compared with those of the plates without the coatings, and coefficient of speed increase was determined. The latter was calculated as a ratio of stability time of MNHP with the coating to stability time of MNHP without the coatings. The tests were carried out in longitudinal sharpening of steel billets of different grades. As prove data obtained, the highest results demonstrated hard alloys after titanizaing in combination with oxidizing and chromization in combination with titanizing. Positive effect of protection coatings is more pronounced on MNHP from the VK8 alloy than from the T15K6 alloy.

CONCLUSIONS

1. Possibility of application on surface of hard VK8 and T15K6 alloys of complex coatings with addition of titanium, chromium, oxygen and nitrogen is shown.

2. Depending upon method of saturation layers on basis of TiC, Cr₂₃C₆ carbides and titanium oxides are formed.

3. Stability of MNHP with the coatings in cutting processing of steels 20, U8A and 40Kh turns out to be higher than that of the initial alloys 1.4–13.5 times.

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Application. Welding and repair in turbine engine fabrication; selection of welding technology for a wide range of workpieces.

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INFLUENCE OF TECHNOLOGICAL FACTORS ON ECONOMIC PARAMETERS OF LIQUID-PHASE REDUCTION

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Results of influence of content of oxide materials in a charge on economic parameters of a melt are presented. It is analytically proved that the main factor, which exerts influence on additional costs in liquid-phase reduction, is content of metal oxides in the charge.

Key words: melt, liquid-phase reduction, metal oxides, scrap, charge, furnace, costs

In recent years intensive works are carried out, directed at study of peculiarities of liquid-phase reduction and finding of technological possibilities of its wide application in production of metal products in metallurgy and machine building [1–3]. Purpose of this work is investigation of peculiarities of physical-chemical processes, which proceed in the molten pool, and determination of main technological factors, which exert decisive influence on economic parameters of the melting process. Peculiarity of the melting consists in the fact that as a charge in melting of a metal oxide-containing materials are used instead of the scrap (primary raw materials, metallurgical slurry and slag, scale, etc.). Their increased content in the charge reduces expenses connected with the charge materials, cost of which exerts significant influence on production cost of the products that creates premises for complete exclusion from the charge of expensive deficient scrap.

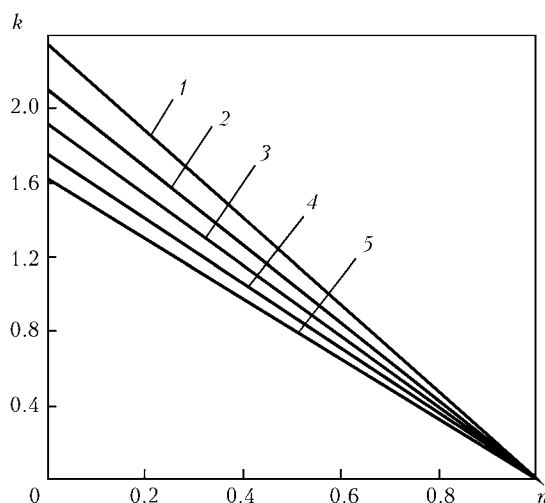


Figure 1. Dependence of coefficient of consumption of wastes k (iron-containing materials) upon coefficient of scrap consumption n : 1 — 45; 2 — 50; 3 — 55; 4 — 60; 5 — 65 % Fe_{gen}

However, replacement of scrap for oxide materials causes additional cost of energy carriers for reduction of metal oxides [4], whereby total cost of the metal melting depends upon content of wastes in the charge, consumption of a reducer, and energy needed for reduction of metal oxides.

Proceeding from this, additional expenses in liquid-phase melting may be presented in the form of the expression

$$E_{add} = G_s \left[C_s \left(\frac{G_s - G_w}{G_s} \right) + C_w \frac{G_w}{G_s} + G_r \frac{G_r}{G_s} C_e \left(1 + q\varphi \frac{G_w}{G_s} \right) \right] \quad (1)$$

or

$$E_{add} = G_s (C_s n + C_w k + m C_r + b C_e), \quad (2)$$

where G_s , G_w and G_r are respectively the content of scrap, wastes and reducer in the charge; C_s , C_w , C_r and C_e are respectively the cost of scrap, wastes, reducer and energy carriers; q is the specific cost of energy for reduction of metal oxides; φ is the degree of reduction of metals; $n = G_s - G_w / G_s$ is the coefficient of scrap consumption; $k = G_w / G_s$ is the coefficient of waste consumption; $m = G_r / G_s$ is the coefficient of carbon consumption; $b = \left(1 + q\varphi \frac{G_w}{G_s} \right)$ is the coefficient of energy consumption.

Analysis of the cost components in the presented expression showed that additional expenses mainly depend upon content of wastes in the charge.

On one hand, increase of content of wastes in the charge causes reduction of energy cost, and, on the other hand, requires for additional expenses for reducer and energy for reduction of metal oxides. It is possible to estimate prevailing influence of separate cost components on additional expenses in liquid-phase reduction and cost of melting as a whole only for conditions of a specific production.

Let us consider, using as an example iron-containing wastes, influence of technological factors on economic parameters of liquid-phase reduction.

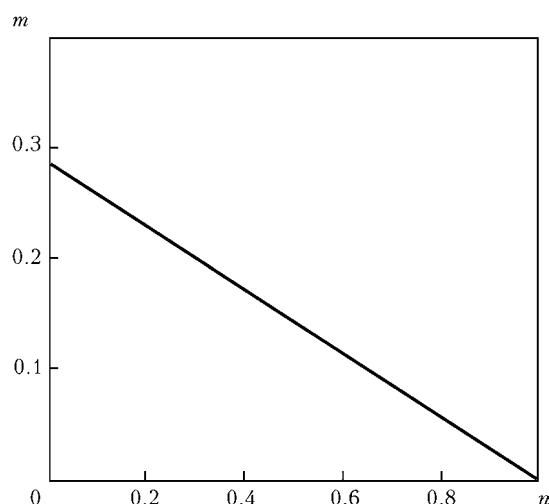


Figure 2. Dependence of coefficient of carbon consumption m upon coefficient of scrap consumption n

In Figure 1 dependence of the waste consumption coefficient upon content of scrap in the charge is shown.

One can see from the presented data that by means of iron content Fe_{gen} reduction in the wastes coefficient of the waste consumption increases. So, at coefficient of scrap consumption n of 0.5, coefficient of waste consumption with content of iron 45 wt. % constitutes 1.18, and at 65 wt.% Fe_{gen} it equals 0.80.

Influence of content of wastes in the charge on coefficient of consumption of carbon, necessary for reduction of iron oxides, is presented in Figure 2.

Analysis of these data showed that by means of reduction of scrap content in the charge coefficient of carbon consumption increases and achieves maximal value in case of complete replacement of scrap for the wastes. This causes increase of carbon content (coke nut, anthracite, etc.) in the charge, which stipulates increase of cost of the charge materials and, as a result, production cost of the metal being melted.

It is known that significant energy is used for reduction of metal oxides in liquid-phase reduction [4]. So, for example, for reduction of iron oxide Fe_2O_3 it is necessary to consume 0.856 kW·h / kg of energy. Such high specific consumption of energy exerts significant influence on its consumption in liquid-phase reduction.

In Figure 3 influence of scrap content in the charge on consumption of energy in liquid-phase reductions is shown. It follows from this data that energy consumption is significantly effected by content of wastes in the charge. By means of the waste content increase consumption of energy grows. When content of wastes achieves 100 %, consumption of energy in comparison

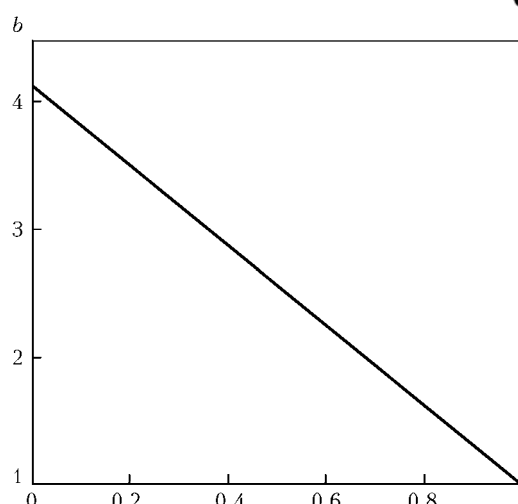


Figure 3. Dependence of coefficient b of energy consumption upon coefficient of scrap consumption n

with conventional arc melting with scrap increases approximately 4 times.

As a rule, liquid-phase reduction is carried out on liquid swamp, for which purpose 20–30 % of scrap of the metal charging is used. Under such conditions of melting consumption of energy will be 3.25–3.5 times higher in comparison with conventional arc melting.

Estimation of the components in expression (1) shows that the greatest influence on energy consumption in liquid-phase reduction is exerts cost of energy carriers and the charge. Carried out calculations allowed establishing that additional cost of the energy carriers insignificantly affect increase of general expenses of metal melting in comparison with scrap melting.

So, data of analytical investigations showed that application of liquid-phase reduction in melting of metal is economically justified. Replacement of the deficient scrap for iron ore materials (ore raw materials, metallurgical slurry, scale, etc.) practically does not increase cost of metal melting in an arc furnace, but improves quality of the metal due to reduction of impurities of non-ferrous metals (chromium, molybdenum, lead, copper, etc.).

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