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PARAMETERS AND CHALLENGES FOR RELIABLE HYDROGEN DETERMINATION IN WELDED JOINTS BY CARRIER GAS HOT EXTRACTION

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ABSTRACT

For the hydrogen-based energy economy of tomorrow, the construction of the necessary infrastructure will play a central role. Most materials used to date, such as welded steels, can be prone to hydrogen embrittlement under certain conditions. This includes the classic delayed cold cracking during welding as well as degradation phenomena during service of components in hydrogen-containing environment. For the evaluation of any hydrogen effect, for example, on the mechanical properties of a welded metallic material, the hydrogen content must be precisely determined. In the case of weld seams, the carrier gas hot extraction (CGHE) according to ISO 3690 is meanwhile state-of-the-art. CGHE is based on accelerated hydrogen degassing due to the thermal activation of hydrogen at elevated temperatures. In addition to the quantification of hydrogen, thermal desorption analysis (TDA) with varying heating rates can be used to determine and evaluate the hydrogen trapping at microstructural defects in the material. For both techniques, experimental and metrological influences must be considered, which have a major effect on the result. For example, ISO 3690 suggests different sample geometries and minimum extraction times for CGHE. This study summarizes the results and experiences of numerous investigations at the Federal Institute for Materials Research and Testing (BAM) with different sample temperatures and geometries (ISO 3690 type B and cylindrical TDA samples) regarding the influence of the sample surface (polished/welded), measurement accuracy depending on the sample volume and the insufficient monitoring of the effect of PI control on the extraction temperature. A deviating extraction temperature from the target temperature can significantly falsify the measurement results. Based on the results, methods are shown which allow the desired extraction temperature to be reached quickly without physically interfering with the measuring equipment. This serves to significantly improve the reliability of the hydrogen measurement through increased signal stability and accelerated hydrogen desorption. In general, an independent temperature measurement with dummy samples is recommended for the heating procedure of choice to exclude possible undesired temperature influences before the measurement. The methods described can be transferred directly to industrial applications.

KEYWORDS: welding, hydrogen measurement, carrier gas hot extraction, ISO 3690, thermal desorption analysis

HYDROGEN MEASUREMENT IN WELDED SAMPLES

For the hydrogen-based economy of tomorrow, the construction of the necessary infrastructure (encompassing transportation and storage) will play an important role. In that connection, joining and welding plays a central role in component manufacturing [1]. For example, the most pipeline materials in the (German) natural gas grid have recently been labeled "H2-ready" [2]. However, a key parameter of degradation by hydrogen, the effective local hydrogen concentration, is not typically quantified and evaluated. Knowledge of this concentration enables the practical evaluation of hydrogen absorption during production or operation. This is particularly important for the welding manufacturing of components, e.g. to avoid hydrogen-assisted cracking or embrittlement. For the determination of hydrogen in a weld seam, the ISO 3690 [3] is a widely applied standard or ANSI/AWS A4.3-93 [4]).

In accordance with reference [3], the mercury method (Hg) and two carrier gas-based methods, gas

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chromatography (GC) and hot extraction (HE), can be used. In GC and HE, a thermal conductivity detector (TCD) is used to quantify hydrogen. The Hg-method is discussed critically regarding health risks and environmental protection and is increasingly being replaced by GC or HE [5]. An additional disadvantage of the Hg-method is the time required for hydrogen collection which takes from 15 to 21 days. With the GC-method, the hydrogen from the welding sample is collected in a closed chamber at elevated temperatures. For this reason, the collection time can be reduced to a few hours [6]. The chamber is then purged with the carrier gas and the corresponding gas mixture is fed into the GC unit. The HE-method is based on the thermal activation of hydrogen atoms in the solid metallic and welded sample, which desorbs and recombines to molecular hydrogen and is collected in a chamber or transported with carrying medium as described in the next sub-section.

Carrier gas hot extraction (CGHE) is a special HE-method. It is characterized by a half-open hydrogen collection chamber that is accessible from one



Figure 1. Carrier gas hot extraction: a — components and working principle of IR-furnace, in accordance with [7]; b — extraction mode 1: Isothermal HE; c — extraction mode 2: HE using variable temperatures/special case of constant heating rates

side only and is continuously purged with an inert carrier gas (e.g., nitrogen). This carrier gas stream transports the desorbed hydrogen to the analyzer. For this purpose, the application of a TCD is defined in ISO 3690 [3]. However, mass spectrometers (MS) are also used now in industrial applications, which offer significantly higher precision. Various heating methods are possible for the CGHE like infrared (IR) radiation, inductive heating, or resistance heating. IR-radiation offers the advantage that the sample can be heated without contact, as well as a wide range of adjustable extraction temperatures and times. Typically, the measured sample itself is not melted if welded samples are investigated [3, 4]. The basic components of the IR extraction system are shown in Figure 1, a. In principle, two operating modes are available, whereas in both cases, the absolute hydrogen concentration can be determined. Mode 1 is shown in Figure 1, b, it encompasses the so-called isothermal HE. This mode is useful for the determination of hydrogen transport data (such as diffusion coefficients) and temperature-dependent trapped hydrogen quantities as shown in [3, 5, 7, 8]. For this purpose, the temperature is kept isothermal during the measurement. Mode 2 is shown in Figure 1, c), it encompasses the HE with variable temperature programs. It is useful to identify the binding energy, the so-called activation energy, of hydrogen at distinct microstructural defects (traps) in the material. For this purpose, sufficiently low heating rates (typically < 0.5 K/s) are applied. The activation energy required to release the hydrogen from the traps is calculated from values obtained using thermal desorption analysis (TDA) [5, 8, 10]. The TDA is also being used more and more in an industrial context but

is currently mostly focused on academic issues (characterization of hydrogen diffusion and trapping).

This study summarizes the results and experience of numerous investigations at the Bundesanstalt für Materialforschung und -prüfung (BAM) with different specimen temperatures and geometries (ISO 3690 type B and cylindrical TDA specimens) for welded samples. For a detailed description, references are given to suitable secondary literature at the relevant positions in this manuscript.

MATERIALS AND METHODS

MATERIALS

The creep-resistant steel T24 //7CrMoVTiB10-10 and the high-strength structural steel S690Q were used for the investigations and welding experiments presented here. Table 1 briefly shows the chemical composition. Further details of the materials can be found in [7]. The chemical composition was determined from an average of five measurements using an optical spark emission spectrometer (from Spectro GmbH).

USED SAMPLE GEOMETRIES AND SPECIFIC HYDROGEN CHARGING CONDITIONS

The first sample geometry was a cylinder (machined from T24) with $\emptyset = 3 \text{ mm}$, 20 mm length and ground surface, see Figure 2, *b*. Some samples were electrochemically charged with hydrogen. For this purpose, a 0.05 M H₂SO₄ acidic aqueous solution with addition of 12 mg/l NaAsO₂ (as recombination inhibitor) was used and various current densities (galvanostatic loading) were applied. After charging with hydrogen, the samples were stored in liquid nitrogen until

 Table 1. Chemical composition of tested materials (in wt.%, Fe — balance)

| Material | С | Cr | Мо | V | Ti | В | Al | Nb | Mn | Si | P+S |
|---|------|------|------|------|------|-------|------|------|------|--------|--------|
| T24 / 7CrMoVTiB10-10 | 0.08 | 2.44 | 1.00 | 0.26 | 0.07 | 0.005 | 0.01 | N/A* | 0.52 | 0.25 | < 0.01 |
| S690Q 0.12 0.50 0.11 0.05 N/A* N/A* 0.01 1.52 0.40 < 0.02 | | | | | | | | | | < 0.02 | |
| $^{*}N/A$ — not available due to measurement uncertainties as impurification in respective steel grade. | | | | | | | | | | | |



Figure 2. IR 07 furnace as part of G4 and G8 analyzer: a — sample position in glass chamber surrounded by IR furnace and magnified view with position of thermocouples inside center position; b — cylindrical TDA sample; c — ISO 3690 type B sample (taken and rearranged from [7], with permission and licensed by Springer Nature)

CGHE. The complete description of the experiment can be found in [8, 10].

The second specimen type was an ISO 3690 type B specimen (machined from S690Q) with dimensions of 30 mm length, 15 mm width and 10 mm thickness, see Figure 2, c. Two representative conditions were investigated to determine the influence of surface finish on temperature development during HE of the ISO Type B specimens: (1) polished/ground surface and (2) in the welded/oxidized condition. For welding, a metal active gas (MAG) welding process with M21 shielding gas (18 % CO₂/82 % Ar) and identical welding consumable was used. The aim of this study was to determine the temperature characteristics during the CGHE, not to determine potential material influences on the hydrogen concentration. However, to maintain realistic conditions, the samples were quenched after welding and stored in liquid nitrogen. For further details on test procedure, we refer to reference [3] and for detailed test results to reference [7].

HYDROGEN

AND TEMPERATURE MEASUREMENT

The CGHE analyzer G4 PHOENIX with TCD was used for the hydrogen determination. For some experiments, a G8 GALILEO (both from Bruker AXS, Germany) with coupled mass spectrometer ESD100 (InProcess Instruments, Germany) was also used. The advantage of both analyzers is that the same IR furnace type (IR07) is used for the HE. For comparison and precision of the measuring methods, we refer to reference [9]. Figure 2, ashows the IR-furnace, with the dashed square indicating the position of the sample during extraction. The center of the longitudinal axis of the sample should always be positioned above the furnace thermocouple. Calibration or dummy samples were used to determine the exact sample temperature. These had identical surface properties and geometry as the later samples for hydrogen measurement. This separation is necessary because an in-situ temperature measurement directly on the sample could falsify the hydrogen content (hydrogen in the thermowire, moist coating, etc.). The cylindrical samples used for the TDA are shown in Figure 2, *b* and the ISO 390 type B sample in Figure 2, *c*. Detailed information on specimen selection and temperature measurement can be found in the reference [7, 8]. As shown in Figure 2, *b* and *c*, an additional blind hole was drilled in the center of the test specimens, into which a type K mantle-thermocouple was inserted. This external thermocouple ensured an independent temperature measurement of the sample bulk temperature and was compared with the adjusted/real furnace temperature. In the case of the ISO 3690 type B specimen, the measurements were taken in the center of the specimen to ensure that the bulk temperature was recorded and not the surface temperature (in case of the isothermal mode 1).

RESULTS

INFLUENCE OF SAMPLE SIZE AND GEOMETRY

In addition to the sample geometry, the sample surface and the ratio to the volume have a significant influence on the extraction temperature or the time to reach the respective temperature. The reason is that the IR-radiation is absorbed at the sample surface. The heating of the material happens due to the heat conduction into the bulk material. For that reason, the temperature change of the sample is primarily influenced by the thermal conductivity coefficient λ (in $W/m \cdot K$) and the thickness of the sample. Figure 3, a shows the ISO 3690 type B and cylinder sample with their heating behavior to 200 and 400 °C respectively (original data shown in references [7, 8]. The desired isothermal extraction temperature (labeled as "furnace)" at the specified temperature is also shown as a comparative value. Of course, smaller samples (such as the cylinders with 3 mm diameter) heat-up faster compared to thicker samples (like the ISO 3690 type B with 10 mm in our case). This applies to both mode 1 "isothermal" and 2 "variable temperature".

Especially for the TDA (using mode 2), an accelerated heating of the sample (i.e., minimizing the tem-



Figure 3. Heating behavior of sample for: a) mode 1 "isothermal holding" for various sample dimensions (taken from reference [7], with permission and licensed by Springer Nature), b) mode 2: measured signal (MS ion current in A) vs. heating rate and deviation between adjusted and measured sample temperature (cylindrical TDA samples), based on [10]

perature gradient in the sample) plays an essential role for reliable desorption curves, see Figure 3, *b*. If the sample temperature is incorrectly assigned, the calculated activation energy can be misleading regarding the share of "diffusible" and "trapped" hydrogen. For example, trapped hydrogen can be detected in low-alloy steels above 100 °C [10]. Only by correctly measuring the temperature directly in or at the sample (i.e., not by an idealized linear heating rate), a realistic activation energy can be calculated. This demonstrates the necessity of using the exact extraction temperature in the sample during the CGHE experiments. This is achieved by previous calibration samples (see Figure 2) without active hydrogen measurement, using the same extraction time-temperature cycle.

INFLUENCE OF SAMPLE SURFACE CONDITION

Figure 4 shows three measured temperature curves of ground (polished) and oxidized ISO 3690 type B samples for a target extraction temperature of 400 °C (according to mode 1). The polished surface ensured



Figure 4. Real heating behavior for mode 1: comparison of a highly reflective and real (oxidized) surface for ISO 3690 type-B sample, targeted extraction temperature 400 °C, (scheme in accordance to [7])

a significant delay in the heating. In this condition, the sample only reaches 350 °C after 1,000 to 1,200 s compared to the oxidized sample with 600 to 620 s. The reason is the different reflectivity (polished steel: 0.1 vs. oxidized: 0.7 to 0.9 in accordance with reference [11]. This means that a polished surface reflects 90 % of the total radiation energy and just 10 % are absorbed, whereas an oxidized surface reflects 10 % to 30 % and absorbs 90 % to 70 %! At constant power level of the IR-radiation emitting furnace, the oxidized sample is therefore heated much faster. Any temperature evaluation of CGHE samples must therefore always be carried out with the identical surface condition that the sample has before the measurement (e.g., oxidized condition for a representative welded ISO 3690 sample or blank metal after electrochemical hydrogen charging). It is therefore generally recommended to carry out an independent temperature measurement by use of the already mentioned calibration or dummy samples (see Sect. 3.1 and shown in Figure 2, b and Figure 2, c), if economically justifiable.

EFFECT OF FURNACE CONTROLLER SETTINGS ON TARGETED TEMPERATURE

The heating process of the sample can be "accelerated" somewhat by changing the heating and preheating settings of the IR07 furnace. The effective controller is of the PID type, whereby the P-component has the greatest effect on the temperature increase. However, this can only be understood in conjunction with the sample geometry. Large samples in relation to the furnace volume, such as ISO 3690 type B, i.e. large sample volumes to be heated, heat up differently compared to the smaller "cylindrical" samples (see Figure 1, *b* and *c*). These react almost instantaneously to temperature changes specified by the heating program, e.g. for mode 1 (isothermal HE) presented in [7, 8] and for mode 2 (variable temperature program/ TDA) in reference [10]. It is therefore not expedient



Figure 5. Accelerated sample heating for mode 1: a — effect of preheating on effusion curve and time "t_{0.5}" after 50 % of the hydrogen is effused from the sample for indented isothermal holding at 200 °C (cylindrical sample), in accordance with [7, 12]; b — schematic representation of furnace preheating before sample insertion on effective sample bulk temperature, in accordance with [8]

to recommend "optimum" controller settings. In addition, the temperature of the sample is ALWAYS below the set one and adjusted targeted extraction temperature of the furnace. The effect on the heating profile of the respective sample geometry should therefore always be checked a-priori using dummy samples. As a best practice recommendation, the desired maximum extraction temperature should be selected 20 K higher. Nevertheless, the ISO 3690 specifies binding minimum extraction temperatures for certain extraction times, but the interpretation is ultimately left to the user. From the point of view of measurement accuracy, a possible difference in the maximum temperature is a matter of interpretation, especially at high temperatures.

EFFECT OF ACCELERATED THERMOSTATING OF IR-FURNACE BY "PREHEATING" ON SAMPLE BULK TEMPERATURE AND CORRESPONDING HYDROGEN EFFUSION

Figure 5, a clearly shows the practical effect of an accelerated heating in mode 1. The measured signal rises much faster in the case of an optimized heating. Figure 5, b exemplarily shows the optimized curve no. "3" (green solid line) compared to the conventional heating procedure shown in curve no. "1" (blue dotted curve) using a preheating concept for the IR-furnace. It is obvious that the sample shows a significantly faster heating. This behavior is particularly important for further considerations of diffusion coefficients. For their calculation, the time " $t_{0.5}$ " is used as standard, at which 50 % of the hydrogen has been extracted from the sample, i.e. 50 % of the respective area of the effusion curve [7, 8, 12]. Using fast heating, the " $t_{0.5}$ " is reached after approx. 50 s at 180 °C, whereas with slow heating it is only reached after at approximately 90 s at 95 °C! This means two things: (1) any calculated diffusion coefficient differs significantly as both " t_{05} " — times differ by factor 2) and (2) the temperature assignment itself would be already incorrect. In any case, accelerated heating is therefore essential to reach the desired extraction temperature. In addition to the controller settings described in Sect. 3.3, this can be achieved very easily by setting a certain preheating temperature of the furnace in advance to the sample insertion. The principle for accelerated heating to different isothermal extraction temperatures is shown in Figure 5, b. For this purpose, the IR07 furnace is preheated to higher temperature than for the hydrogen extraction needed. From the moment the sample is inserted, the IR-furnace temperature drops freely as an exponential function in accordance with the thermodynamic inertia of the "furnace with sample" system. As a result, considerably more energy is available for the sample, as the furnace itself must not be additionally heated up during the isothermal hydrogen extraction. Further details were presented e.g. in [7, 8, 12].

CALCULATION OF RELIABLE HYDROGEN CONCENTRATIONS WITH AUTOMATED SOFTWARE SETTINGS

Typically, the total hydrogen concentration is calculated by the integrated measured signal over time (e.g., in mV for the TCD or in A as ion current for the MS) using a calibration factor and related to a certain sample mass. The most frequently used unit is "ml/100 g Fe". This means a certain volume of hydrogen dissolved in a virtual deposited metal of 100 g. An essential factor for reliable measurements is the human being in terms of literally "blind trust" in automatically generated values. This applies regardless of the manufacturer. The extraction time and the temperature program for mode 1 or 2 are usually set by



Figure 6. Possible errors when evaluating the measurement signal: a — extraction time selected too short; b — incorrect settings for integration limits or insufficient knowledge of signal switch-off threshold, see [7]

humans, but the total hydrogen concentration " HD_{tot} " is usually calculated automatically (by the respective controller software). Two effects can occur here:

At first, an insufficient extraction time (e.g., using mode 1). This is usually the case if the hydrogen measurement is carried out with a maximum pre-set holding time, for example in accordance with ISO 3690 [3] as shown in Figure 6, *a*. In the case of 400 °C, an extraction time of 21 minutes is recommended in the standard. If this time is set as the maximum extraction time or the measurement is aborted before the total hydrogen "HD_{tot}" effused from the sample, this results in an error due to the "not detected" hydrogen (gray shaded area and labeled as "HD_{ND}"). In addition, only a sufficient extraction time duration at the desired temperature ensures that the total hydrogen "HD_{tot}" can desorb from the sample and be measured by either TCD, MS or others.

Secondly, the determination of " HD_{tot} " is mostly based on the pre-set start and end points (by certain signal cut-off thresholds) to automatically calculate the detected hydrogen. The baseline is usually set by the software itself, based on the lower signal integration limits (in the case of a TCD, set as the mV threshold value in the control software). This cut-off limit is the most important limit for the integration limit of the signal, i.e. the amount of hydrogen extracted from the sample. This is particularly the case for long desorption/extraction times with a low signal-to-noise ratio. For very long measurements/very low hydrogen effusion rates, the hydrogen detector (TCD or MS) is virtually operated at the detection limit. It is always advisable to check the measurement curves afterwards manually and ensure that the integration limits are set precisely. Especially for small amounts of hydrogen dissolved in small samples, it may be recommended to switch to a measuring system with higher precision such as a MS. As shown in Figure 6a, errors in

the evaluation (i.e., underestimation of the calculated "HD_{tot}") may otherwise occur. In this case, a part of the hydrogen quantity present in the sample would not be detected (gray shaded area "HD_{ND}" in Figure 6, *b*). This was also the subject of controversial discussions at a regular review of ISO 3690 [13, 14]. For further details on these extreme cases discussed in this sections (including the assessment of signal quality) can be found in the references [7, 8].

CONCLUSIONS FOR A RELIABLE HYDROGEN DETERMINATION VIA CGHE

The following chapter briefly summarizes the results described. It is important to note that additional special metrological specifics apply when using an IR furnace for extraction. These are:

The IR radiation is absorbed at the sample surface. This process depends strongly on the surface conditions of the sample, i.e. polished/ground or oxidized and is expressed by the absorption or emission coefficient.

At constant power of the radiation source, a larger proportion of reflected radiation leads to delayed heating of the sample to the desired extraction temperature. This effect intensifies with greater thickness. The measurement time required for hydrogen extraction is therefore directly linked to the heating behavior of the sample to the desired temperature.

The sample is heated exclusively by heat conduction from the surface to the interior, which is characterized by the specific thermal conductivity. This means that there is ALWAYS a temperature deviation between the set furnace temperature and the temperature of the sample due to the system. For a pure hydrogen determination according to mode 1, this behavior is secondary, but for the evaluation of the diffusion behavior mode 2 is essential.

Materials with higher thermal conductivity (such as copper) could be heated faster compared

| Influence | Effect | Countermeasure | | | | | |
|-------------------------------|---|---|--|--|--|--|--|
| Sample geometry | "Thermodynamic inertia" → maximum IR-furnace power limits the heating efficiency of the samples Thicker samples require prolonged time for heating to elevated temperature | Mode 2: Prefer thinner samples for TDA Use of dummy samples and recording of temperature development Restriction to "known geometries, e.g. according to ISO 3690 | | | | | |
| Sample surface | IR radiation is absorbed at sample surface Heating of bulk via heat conduction → delay and temperature gradient Blank metal surfaces heat up slowly Highly reflective materials have a limited suitability for application of CGHE | Adjust surface to the desired heating mode Application of mode 1: Determination of hydrogen concentration (e.g., according to ISO 3690) or diffusion coefficients Application of mode 2: Electrochemical and/or pressurized hydrogen-loaded samples Consideration of the influence especially for TDA (temperature offset) Use of dummy samples and recording of temperature development (determination of temperature off-set) | | | | | |
| Temperature monitoring | Difference of sample bulk and surface temperature Occurrence of unavoidable but limitable temperature gradient | Sample position in extraction chamber should be identical with IR-furnace thermocouple Use of dummy samples Recording of temperature profiles (determination of temperature offset) | | | | | |
| Settings of PID-controller | • Consideration of the heating behavior of the samples and effect on ex-traction behavior | "Calibration" of representative temperature curves and optimization of the setting values "Preheating" of IR-furnace to accelerate sample heating | | | | | |
| Quantitative measurement | Precision of the measuring system vs. effusion rate Consideration of signal stability (signal-to-noise ratio) TCD vs. MS | MS enables use of smaller sample geometries and weight Better measurement resolution and sensitivity | | | | | |
| Hydrogen concentration | Observing the signal thresholdsCorrect setting of integration limits | • Critical assessment of results ("visual inspection" of measured values) | | | | | |

| Table 2. | Expe | erimental | and and | alyzer | · influence | on l | hydrogen | detection | and | countermeasures | for | limita | tion |
|----------|------|-----------|---------|--------|-------------|------|----------|-----------|-----|-----------------|-----|--------|------|
| | | | | | | | J D - | | | | | | |

Table 3. Material effect on hydrogen detection and countermeasures for limitation

| Influence | Effect | Countermeasure |
|----------------------|--|---|
| Micro- structure | Austenite with increased hydrogen solubility compared to Ferrite Diffusion in Austenite decreased | Variation of extraction time necessary Avoidance of underestimation of hydrogen concentration → Consideration of dummy samples In case of unknown materials → "Trial and error" |
| Chemical composition | "The more complex the alloy, the more complex the hydrogen trapping" Shift of desorption temperatures/time "Unknown" materials require special attention | Variation of extraction time necessary Consideration of dummy samples In case of unknown materials → "Trial and error" |

to iron or steel, but the high reflectivity is usually in contrast to this.

The effect of reaching the isothermal extraction temperature over time (mode 1) is of secondary interest if the extraction time is sufficiently long (i.e., only determination of the water content is of interest). As already mentioned, the temporal interpretation of the heating behavior (and thus the correct temperature assignment) in mode 2 is essential. This is the only way to correctly calculate the activation energy of the hydrogen traps via the TDA. The facts described above provide options for influencing the HE without having to physically intervene in the process. Table 2 briefly presents the main influences presented in Sect. 3.1 to 3.5 and derives measures to limit them (shown in bold).

The countermeasures presented in Table 2 allow the desired extraction temperature to be reached without physically interfering with the measuring equipment. They can be directly transferred to industrial applications by adapting the software of the analyzer (further details can be found in [7, 8]). Table 3 gives additional examples of issues to be considered when comparing different metallic materials or processing conditions. Especially, "new" or unknown materials require special attention [15].

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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