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Instrumented indentation at elevated temperatures for determination of material properties of fusion relevant materials

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ABSTRACT

The testing of small sized samples is an important advantage of the instrumented indentation with respect to the investigation of materials for fusion application. A continuous recording of the indentation depth and force enables a determination of mechanical properties of the tested material.

In this study, the results of the high temperature experiments with a custom made indentation device are presented. The reduced activation ferritic martensitic steel EUROFER is investigated in an unirradiated state with spherical tips and for the first time Vickers tips at increasing temperatures up to 500 °C.

The indentation procedure is numerically simulated at different temperatures and the corresponding load-displacement-data are compared with the experimental results. A quantification of the influence of variations of the indentation tip radius is presented as well.

Finally, the operation of the indentation device with respect to the restrictions of the Hot Cell environment is discussed.

1. Introduction

The development and qualification of new compositions of structural materials for the application in future fusion power plants is an important field of current fusion research. Hence, the comprehensive characterization of such materials at their operation conditions is an indispensable part of the work of fusion material scientists all over the world. For the investigation of neutron-irradiated materials, the instrumented indentation is an attractive and promising method. The use of small sized indentation samples has a positive effect on the costs of irradiation programs and the dose rate of the single specimen and is indispensable for a future neutron source, because of its small irradiation chamber. With respect to the Hot Cell environment, the relatively simple preparation of the samples simplifies a multiple testing. Therefore, a maximum of information can be obtained from the investigated material.

Instrumented Indentation at elevated temperatures is an important research field with remarkable progresses in the recent years. For example the evaluation of welded materials, nanoindentation at high temperatures, and the investigation of different structures and materials are part of current research, e.g. [1–4]. Especially with respect to investigations related to nuclear fusion, successful work has been done with high temperature indentation experiments [5] and investigations of ion implanted thin layers using nanoindentation [6, 7]. A further possible application could be the investigation of materials with respect to the ductile-to-brittle transition behavior [8–10].

In a fusion reactor, the structural materials have to withstand extreme loads due to the nuclear fusion process, like high neutron radiation and high temperature conditions. A candidate material like EUROFER, a low activation steel, needs to be investigated in irradiated state at the operation conditions of nuclear fusion, to obtain a complete understanding of the material behavior [11].

By using a commercial indentation system at the Fusion Material Laboratory (FML), promising results already were obtained by instrumented indentation experiments at room temperature on irradiated specimens [12].

For investigations at elevated temperatures, a new indentation device was developed at the Karlsruhe Institute of Technology (KIT). In contrast to commercial systems, the device is designed for future remote-handled investigations of neutron irradiated materials in a Hot Cell of the Fusion Materials Laboratory at KIT. The machine enables indentation experiments at temperatures up to 650 °C with a maximal testing force of 200 N [13, 14]. First results of high temperature indentation tests are shown in [15].

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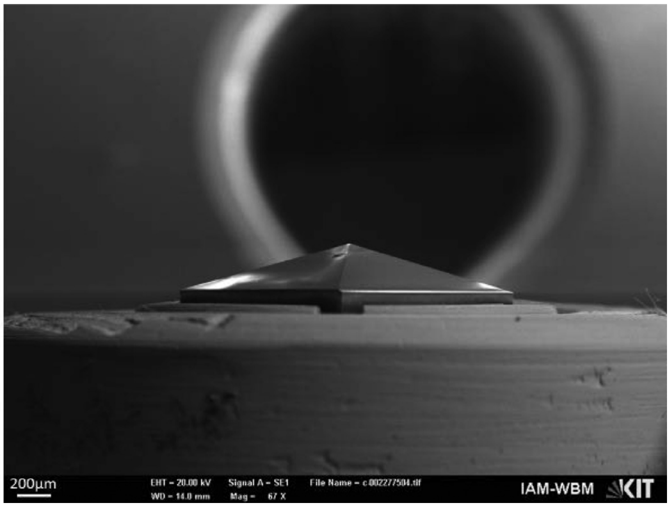

Fig. 1. SEM micrograph of an indenter consisting of a TZM holder and a Vickers diamond tip.

2. Experimental

2.1. Indentation device

The indentation column with the integrated indenter, the sample stage and the heating system are installed inside a vacuum chamber to prevent an oxidation of indenter and sample. The indentation depth measurement system is located outside of the vacuum chamber. The indentation experiments are performed in force control.

The sample and the indenter tip are heated up separately via independent heating cartridges, which are located inside the indentation column and the sample stage. The active temperature regulation uses the signals of a pair of thermocouples. The high temperatures up to 650 °C necessitate a water cooling system. Hence, stable thermal conditions are achieved for the sample and the indenter tip.

For the measurement of the indentation depth, an optical system is used, because of its insensibility to the temperature conditions inside the vacuum chamber. Using image capturing, via a camera in combination with a long distance microscope, and image correlation in post-process, it is possible to determine the indentation depth from the relative movement of the sample and the indenter. The two measurement points of the optical system are located very close to the contact point of tip and sample. Hence, the influence of thermal expansion on the measurement is minimized.

In combination with the force measurement, the load-displacement-curves could be determined for every indentation experiment by using a custom made software with a resolution of 0.1 µm and 0.05 N for the displacement and the force, respectively.

For the use of the high temperature device, custom made indenters are necessary. These indenters consist of a titan zirconium molybdenum (TZM) holder and the tip. In the present study, indenter with rounded Rockwell cones with a radius of 200 µm and pyramidal Vickers tips are used, see Fig. 1. Diamond and sapphire are used as tip materials. A mechanical clamping system fixes the diamond tip in the holder. The pressure sensitivity of sapphire does not allow the use of this system for the sapphire tips. Therefore, an aluminum nitride ceramic adhesive with an application temperature of 3000 °C is used for the fixing of the tip.

All indenters are designed in a way that they can be handled with manipulators in a Hot Cell to enable the use of different tip geometries and the exchange of broken indenters.

More detailed insight in the indentation device and the different setups are given in [14] and [15].

2.2. Material and samples

The tested material EUROFER (9CrWVTa), a customized ferritic martensitic steel, is a specially developed alloy for fusion application. The reduced activation of the material is achieved by substitution of high activation alloy components, e.g. Mo, Nb and Ni, by elements with lower activation, e.g. W, V and Ta, [16]. The heat treatment of the material was 1040 °C for 0.5 h and 760 °C for 1.5 h.

As specimens, broken halves of Charpy impact or mini fracture mechanics tests with a size up to $15 \times 6 \times 3$ mm³ have been used for the indentation experiments. As preparation, the samples were multi-level grinded and subsequently polished with a finish of 3 µm diamond paste.

The indentation experiments are evaluated according to Brinell and Vickers on basis of an optical measurement of the diameter of the indents via an optical microscope according to Brinell and Vickers, DIN EN ISO 6507 and DIN EN ISO 6507.

Additionally, the load-displacement-curves of the individual experiments are determined and evaluated, according to the standard DIN EN ISO 14577. Mono cyclic experiments with a maximal load of 40 N, a loading rate of 1 N/s and a holding time of 15 s were carried out.

All the experiments were carried out between room temperature and 500 °C with spherical and Vickers indenters with diamond and sapphire as tip materials. The indentation depth of all presented experiments exceed the threshold value of 6 µm stated in DIN EN ISO 1477.

The upper temperature threshold for diamond is 400 °C, because of its increasing chemical instability in contact with steel at elevated temperatures. Further information of indenter tip material behavior at elevated temperatures is given in [17]. The vacuum pressure during the experiments was between 8×10^{-6} mbar to 1×10^{-5} mbar.

Due to the limited sample size and the wide range of test temperatures, a multiple indentation at every test temperature was not possible.

3. Results and discussion

3.1. Brinell and Vickers hardness

In this section, the conventional hardness according to Vickers and Brinell are evaluated. In Fig 2, the Brinell hardness (HB), the Vickers hardness (HV) and the ultimate tensile strength (UTS) [11] are plotted vs. the temperature. The results for both sapphire tip geometries exhibit the same temperature dependency of the material hardness of EUROFER and can be verified with the tensile test results (UTS). The hardness decreases continuously with an increasing test temperature until 400 °C. Between 400 °C and 500 °C, the drop of the hardness is stronger.

A comparison with the results of the investigations of MANET II in [15], which were carried out with a diamond tip, shows the practicability, on the one hand of sapphire as tip material and on the other hand of the whole design of the custom made indenters for different tip geometries. Indenter with cube corner tips are already produced and corresponding investigations are planned.

In Fig. 3, the HV of EUROFER is plotted for the diamond and the sapphire tip and a clear variation of the hardness values is visible over the whole testing temperature range. The results determined with the sapphire tip are about 1.5% higher than those obtained with the diamond tip. A similar phenomenon was discovered in a measurement campaign on MANET II with spherical indenter tips, see [15].

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