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Microcharacterization of damage in materials for advanced nuclear fission plants

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Abstract. Miniature and sub-miniature samples were used for determination of mechanical properties of materials for advanced fission plants. Results from indentation and focused ion beam prepared micro-samples, punch tests and thin strip (irradiation) creep tests are shown. The results allow conclusions concerning materials damage. Irradiation damage profiles were determined with indentation. Results from micro-pillar tests showed a good agreement with results from conventional samples in case of oxide dispersion strengthened steels. Thin strip irradiation creep experiments revealed a negligible influence of dispersoid size/distribution on creep rates. Punch tests of fibre reinforced materials showed consistent results which still need quantitative analysis.

Introduction

Damage assessment in components for advanced nuclear plants (e.g. gas cooled reactors) requires information about degradation of mechanical properties and its link to microstructural changes. Miniature and sub-miniature probing is an important tool in this respect. Such techniques allow determination of properties from small volumes which is important for nuclear materials mainly due to two reasons: The total activity of activated samples can be kept low, on the one hand. Ion implantation is commonly used to study irradiation effects. However, the limited penetration depth of ions necessitates analysis of small damaged areas, on the other hand. Design of advanced fission and fusion plants is only possible with materials which are able to operate at high temperatures, high flux and in hostile environment. Oxide dispersion strengthened (ODS) steels and SiC compounds belong to such materials. Different types of ferritic oxide dispersion strengthened (ODS) materials and SiC and C fibre reinforced SiC samples are investigated within our EXTREMAT project. Some results gained on these materials will be discussed in the following.

Materials and irradiation

The commercial FeCrAl ODS alloy PM 2000 [1] was analyzed in the annealed condition and after severe plastic deformation (SPD) by Equal Channel Angular pressing (ECAP) [2]. Additionally, samples with very small (few nano-meters) dispersoids (ODS 19Cr steel) [3] were tested. Different qualities of SiC_f/SiC and C_f/SiC were tested as potential candidates for control rods in very high temperature reactors (VHTR). The SPD material was supplied by the EXTREMAT partner ARCS. We investigated four qualities of reinforced ceramics: chemically vapour infiltrated (CVI) SiC_f/SiC 2D-grid, (1417_II),CVI C_f/SiC 2D- grid, (5054_II) both from MAN Technology (MT-Aerospace), liquid silicon infiltrated (LSI)C_f/SiC 2D-grid (PH970 P), LSI C_f/SiC irregular (HP164P), both from DLR.

Ion implantation was performed at room temperature with a $^{4}He^{++}$ beam at the Swiss Federal Institute of Technology in Zürich, using a Tandem Accelerator. To obtain an acceptable damage distribution irradiation was performed under 4 different incident angles (ranging from 0° to 66°) and an energy of 1.5 MeV. Damage as a function of depth ranged from 0.7 dpa at 1 micrometer to about

1.3 dpa at 2.5 µm. More details about irradiation and profile can be found in the literature [4]. *In situ (irradiation) creep* under He-implantation was performed at the compact cyclotron of Forschungszentrum Juelich. Details of the experimental set up are described in [5]. With 24 MeV $^4He^{2+}$ ions passing through a magnet scanning system and a degrader wheel with 24 Al-foils of variable thicknesses, the 0.1 mm thick samples were 3D-homogeneously irradiated under constant uniaxial stress. *Thermal creep tests* were performed with the same sample geometry, however, outside of the cyclotron. More details about the experimental conditions can be found in literature [6].*Microhardness and indentation tests* were performed on surfaces polished prior to irradiation. *Pillars* were produced in two steps using a focused ion beam: Coarse – 30 keV Ga ion source, 25.1 keV Condenser, 400 µm aperture, 50 nm spot beam, 4 µm pillars diameter, Fine – 30 keV Ga ion source, 24 keV Condenser, 50 µm aperture, 10 nm spot beam, 1 µm pillars diameter . *Punch tests* were done with different tool geometries (sphere, cyclinder) of different sizes (1mm, 3mm, 5mm). Damage was also characterized with TEM and with advanced beamline techniques. The paper will summarize important results and discuss them with respect to their relevance for larger volumes.

Microhardness and indentation

Microhardness testing is a well established method for assessment of the yield stress of materials. It can therefore be conveniently used for determination of hardening or softening. Instrumented hardness testing (micro/nano-indentation) underwent a strong development over the past 15 years and it allows today quantitative conclusions about yielding (particularly with finite element support). Relative differences of microhardness were found comparable with the expected changes in yield stress [7], also in the tests performed with the three qualities of ODS steels. A very good example of the detection of uneven distribution of irradiation damage is given in Figure 1. Annealed PM2000 and SPD PM2000 samples were He-ion irradiated and a hardness-depth profile was measured with a nano-indenter running continuous stiffness mode. The measurements were compared with the damage profile calculated with SRIM [8].

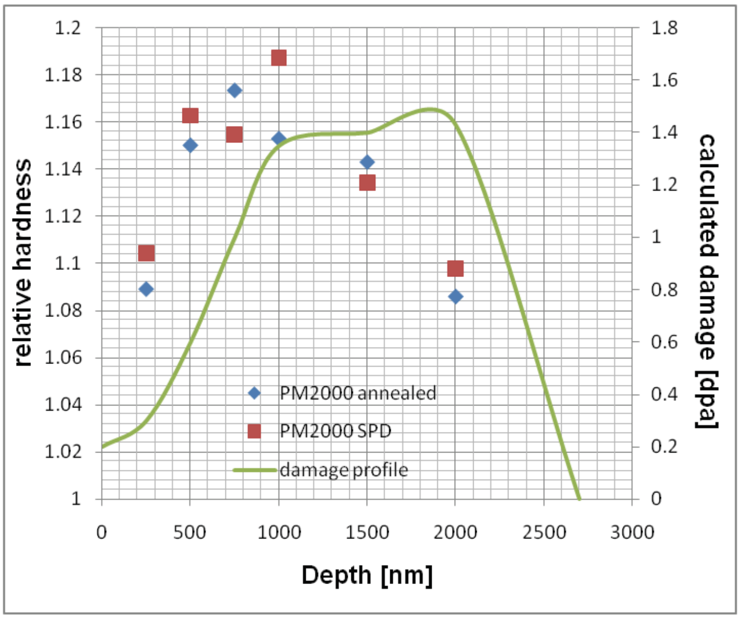

Figure 1: Irradiation hardening profile of two qualities of He-implanted PM2000 samples. The calculated damage profile is shown for comparison

Two conclusions can be drawn from Figure 1. There is no difference between the annealed material and the SPD material and the damage profile is quite well reproduced. The shift of about 500 nm between the calculated and the measured profiles can be explained by the fact that the indentation "senses" information from areas being ahead of the tip. The relative hardness in Figure 1 is calculated as the ratio of the hardness values measured before and after irradiation. It is thereby assumed that surface effects cancel out.

Small punch tests

Samples of the size of only a TEM-disk are required to get information on strength and yielding behavior of metallic materials with small punch tests. Correlation of stress-strain curves with results of small punch tests can be obtained for metals by finite element analysis (see e.g. [9]). Less information about the applicability of such tests is available for non-metallic materials like fibre-reinforced ceramics. As C_f/SiC and SiC_f/SiC materials are an important class for future gas cooled reactors and fusion applications the punch test could be suitable for determination of irradiation damage also for this class of materials. In contrast to metals which can be considered as elastic-plastic continua this is no longer true for fibre reinforced materials. Influences of sample diameter and tool sizes are expected to become important. As some sample features reach dimensions, comparable with the testing device , it is also expected that variations could arise from different testing locations. Figure 2 summarizes the mean values of the maximum stresses determined with different tools normalized to the material quality of the CVI C_f/SiC material 1417 II. Although the analysis of the results has not yet been finished and no quantitative conclusions can therefore be drawn, it seems that the relative strengths could be reproduced by the different methods. In general it was found that the scatter of the data is very high, making testing of many samples necessary.

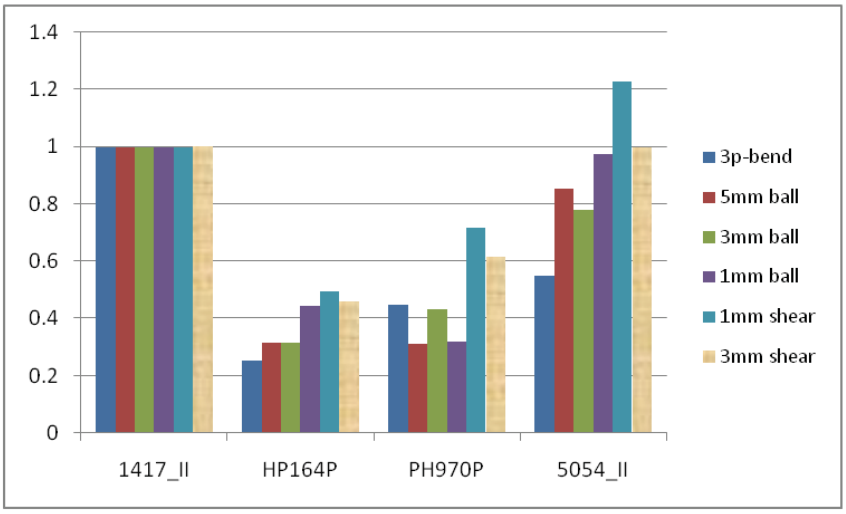

Figure 3: Comparison of maximum stresses determined for different qualities of reinforced SiC matrix materials with punch tests and 3-point bend tests normalized the material quality of the CVI C_f/SiC material 1417 II

Use of punch test could be advantageous, particularly when only a limited amount of sample material is available.

Micropillars

More quantitative stress-strain data can be obtained with micro-samples prepared by focused ion beam (FIB). Micropillars, bendbars and other sample geometries are employed to study mechanical properties of micrometer cube volumes. The influence of sample dimensions is certainly a critical issue (e.g. [10]). In case of annealed PM2000 no size effects were found with micro-pillars of 1 micrometer diameter [7], as shown in Figure 3. This fact was attributed to the presence of

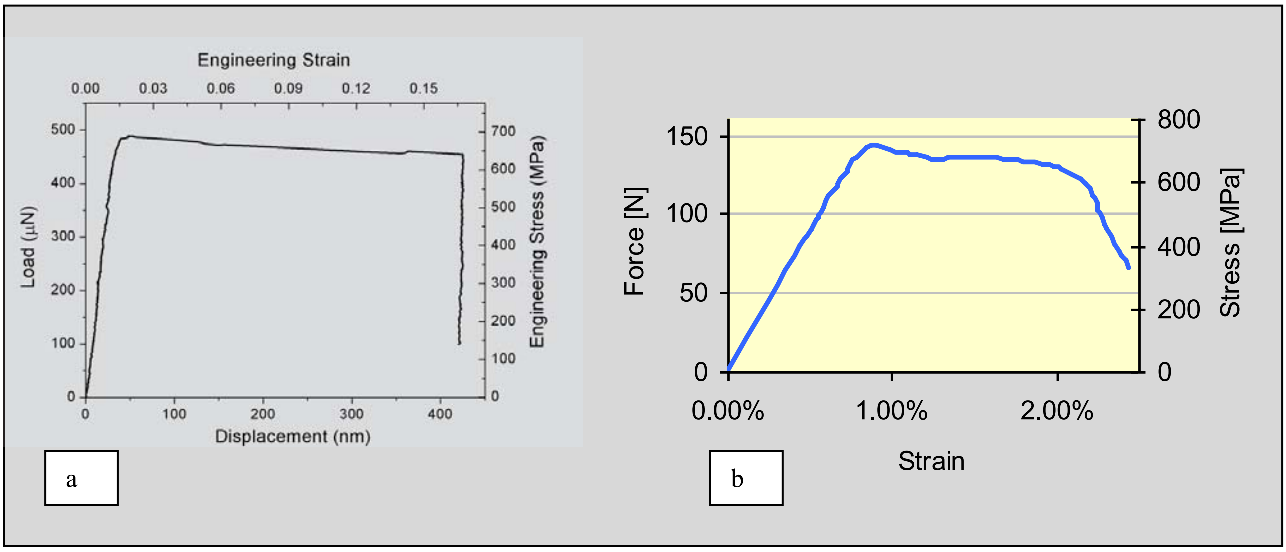

Figure 2: Comparison of stress-strain responses of a micro-pillar sample (a) and a non-micrometer tensile sample (b). Material: annealed PM2000.

dislocations in the pillar, the presence of dispersoids and the fact that single crystal conditions were investigated (no grain size effects).

Thin strip tensile/creep testing

Determination of creep under elevated temperature and irradiation is expensive and time consuming when performed in reactors. Tests under ion irradiation can be done in accelerators. However, the limited penetration depth of the ions limits the thickness of gage sections to 100-200 micrometers. It could be shown already that steady state creep rates determined with such thin strips are in good agreement with the expectations from conventional samples [11]. Differences might exist for the third creep stage which could have an impact on stress rupture and rupture elongation results. Irradiation and thermal creep experiments were performed with annealed PM 2000 and with the nano-particle ODS 19Cr material. An almost temperature independent compliance of $5.7 \cdot 10^{-6}$ $dpa^{-1}MPa^{-1}$ was determined for annealed PM2000. Similar results have been obtained for ODS 19Cr containing ODS particles with one order of magnitude smaller diameter. Size of dispersoids and their distribution which are the main microstructural elements responsible for the excellent high temperature creep resistance of ODS alloys play obviously only a negligible role in irradiation creep.

Conclusions

The capabilities of different types of sub-sized samples (indentation, micro-pillars, small punch, thin strips) for determination of mechanical properties of advanced gas cooled reactor materials were investigated. Different types of ODS materials and of fiber reinforced SiC were used. Indentation could not only be used to determine the yield strength of metallic materials. Irradiation damage profiles could be also determined. FIB-prepared micro-samples are expected to have a high potential for determination of local mechanical properties. For large grained annealed PM2000 samples no pronounced size effect on the shear stress was measured, which is not considered as a general behavior. Small punch tests on reinforced SiC materials lead to a ranking of materials which is comparable to that obtained with 3-point bend tests. A quantitative assessment of the use of punch tests results for such materials is still lacking. Thin strip in situ irradiation creep tests on ODS materials did not show a significant influence of dispersoid size on creep rates. In summary, it can be stated that currently available sub-sized specimens and micro-testing methods are well suited for the analysis of damage in materials for future advanced nuclear plants.

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