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The effect of pre-deformation on the ductility of chromium

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Abstract

Pure chromium produced via a powder metallurgical route exhibits brittleness at room temperature (RT) in the recrystallized condition. The ductile to brittle transition temperature (DBTT) lies between 220 °C (bending and tensile tests) and 300 °C (fracture toughness experiments). To increase the ductility at RT the effect of plastic pre-deformation has been investigated. Small pre-deformations above the DBTT result in ductility in bending and in tension at RT. Severe plastic deformation (SPD) by equal channel angular extrusion and by a special developed procedure leads to a significant increase in both in strength and in ductility. However it is important to avoid the nucleation of small cracks during SPD in order to achieve a very good ductility.

1. Introduction

Due to their low neutron-induced radioactivity, chromium-based materials are considered to be candidates as structure materials in fusion technology. Drawbacks for the application of these materials in industrial design are their brittleness at room temperature (RT) and their high ductile to brittle transition temperatures (DBTT). In this paper, mechanical and fractographical investigations are presented of pure chromium (DUCROPUR) with a purity of about 99.97% (N: 19 μg/g, C: 51 μg/g, O: 40 μg/g, H: 2 μg/g). The material has been produced through a powder metallurgical route by the company Plansee and has been deformed using different procedures.

2. Results of chromium in the recrystallized condition

The mean grain size of DUCROPUR in the recrystallized condition (DP-R) is about 80 μm.

The dependence of bending fracture strength σ_F and bending 'yield strength' σ_y (plastic deflection of 0.01 mm) on temperature is depicted in Fig. 1. Below 220 °C the specimens show no macroscopic plastic deflections. Above 250 °C large plastic deflections are obtained in bending as well as in tension. The DBTT in bending and in tension lies between 220 and 250 °C.

In the fracture toughness investigations, a somewhat higher DBTT is observed. Fracture toughness increases only slightly from 7.7 MPa $\sqrt{m}$ at RT to 17.5 MPa $\sqrt{m}$ at 290 °C. Above 300 °C the fracture behavior changes rapidly and dramatically. At 320 °C the samples show large plastic deformations at the crack tip before final fracture (critical crack tip opening displacement is about 1.4 mm) and the toughness value calculated from a J-integral test is about 500 MPa $\sqrt{m}$.

A detailed presentation of the mechanical behavior of DP-R is given in [1].

3. Results of chromium pre-deformed in bending and in tension

Bending samples (cross-section 6 × 6 mm) of DP-R have been plastically pre-deformed at 400 °C (plastic deflection about 1 mm at a span width of 24 mm) and tested at RT. In contrast to the tests with DP-R the

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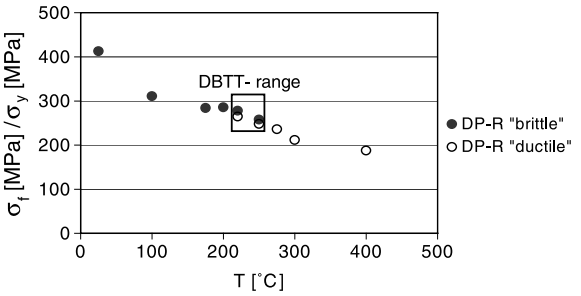

Fig. 1. Dependence of bending strengths on temperature; the full symbols indicate fracture strengths (σ_F), the open symbols indicate 'yield strengths' (σ_y) at the plastic deflection of 0.01 mm (specimen dimensions 6×6 mm, span width 24 mm).

pre-deformed samples show ductility at RT. A bending angle of 10° is obtained before the final fracture occurs in a brittle mode.

Similar to the pre-deformation in bending, tensile samples of DP-R have been pre-deformed ($\varepsilon_{pl} = 3\%$, 5% or 10%) at about 300 °C. The stress–strain curve at this temperature indicates a pronounced yield point and an area of 'Lüders strain'. This indicates a significant amount of interstitial solved impurities. All pre-deformed samples show again ductility at RT. For example a 5% pre-deformed specimen at 300 °C exhibits at RT a plastic strain of about 6.5% before the brittle final fracture occurs.

4. Chromium deformed by severe plastic deformation

DP-R has been deformed by equal channel angular extrusion (ECAE) [2] at a temperature of about 320 °C. The channel angle was 120° which corresponds to a true strain of $\phi \approx 1$ per pass. The friction between the ECAE device and the DP block is the reason why a multiple pass deformation in our experiment failed until now. After a single pass cracks have generated on the sample surfaces and an additional deformation pass results in the breaking of the specimen.

In order to reach a larger degree of deformation and a characteristic 'severe plastic deformation (SPD) microstructure' a special developed SPD process has been applied [3]. The deformations have been performed at RT to a true strain of $\phi \approx 0.69$ and between 350 and 400 °C to true strains of $\phi \approx 2.77$ and 5.55.

From the resulting blocks which have been deformed by both ECAE and the special developed SPD process bending samples were machined to investigate the influence of deformation on strength and ductility.

To investigate the influence of even larger degrees of deformation on the microstructure high pressure torsion (HPT) experiments [2] have been carried out. The applied stress during HPT was 3 GPa and the deformation temperature lied between 320 and 400 °C.

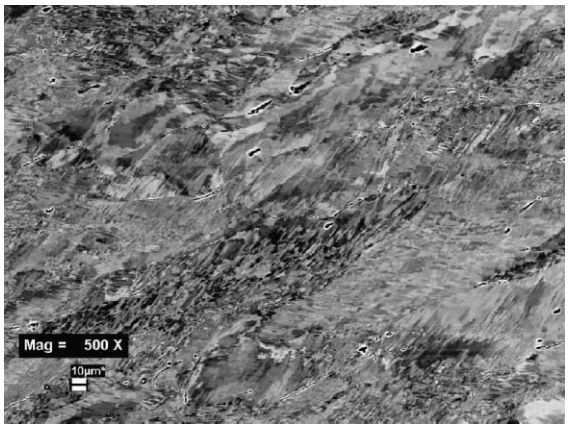

Fig. 2. Electron back scatter micrograph of DP after deformation by single passed ECAE.

4.1. Developed microstructure by severe plastic deformation of pure chromium

During all SPD processes (at RT and above the DBTT) small cracks have nucleated, where the number and size of cracks depend on the deformation process, temperature and true strain. A scanning electron microscope (SEM) micrograph of the microstructure after a single pass ECAE deformation is depicted in Fig. 2. It seems that the already existing sinter pores are responsible for this crack nucleation. Fig. 2 shows further the well developed cell or cell block structure of the microstructure.

Electron back scattering diffraction (EBSD) measurements [4] have been performed to investigate the changes in microstructure during deformation. Fig. 3 displays the data directly obtained by the EBSD measurements. The maps of the inverse pole figures (IPFs) display the contribution of the crystallographic orientation of chromium after the different deformation processes and different degrees of plastic deformation. The points where the EBSD system could not determine an orientation due to a bad quality of the diffraction patterns or an overlapping of patterns from differently oriented regions are marked with black. Also due to the bad pattern qualities, the orientation of few isolated points may be determined incorrectly. Furthermore the misorientation between two adjacent regions has been determined by using an automatic evaluation program.

It is observed that after a deformation degree of $\phi \approx 2.77$ in the original grains, the formation of cell structures sets in. Large angle grain boundaries are only observed between the original grains. This cell structure

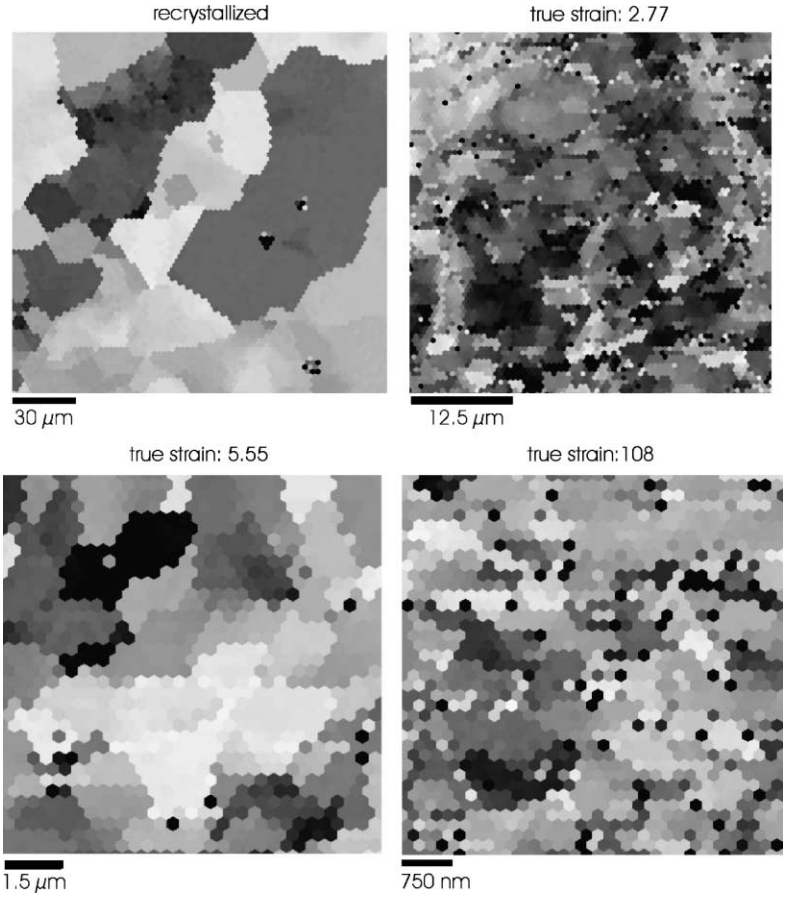

Fig. 3. IPF maps of DP in the recrystallized condition and after a deformation of $\phi \approx 2.77$, 5.55 and 108 (unfortunately the colored maps can here presented only in gray which gives only a qualitative impression of the developed orientation distribution).

can also be observed after a deformation of $\phi \approx 5.55$ but here also the formation of cells or cell blocks (or sub grains) with a misorientation larger than 10° and a largest size of 2 μm has taken place.

In Fig. 4 an electron back scatter micrograph of the microstructure deformed by HPT ($\phi \approx 108$) is depicted. A very fine grained equiaxed microstructure can be observed. The EBSD measurements verified that the observed regions have a misorientation greater than 10°. A new microstructure with a grain size significantly smaller than 1 μm has been developed.

4.2. Mechanical behavior of deformed chromium by severe plastic deformation

The bending fracture strengths at RT of samples deformed by a single passed ECAE ($\phi \approx 1$) and by the special process ($\phi \approx 0.69$) are about two times larger than those of DP-R. Furthermore the samples show ductile behavior (bending angle ≈2°). However it should be noted that these samples contained a large number of microcracks. It seems that it is possible to obtain larger bending angles when it is possible to prevent crack nucleation during SPD because these cracks act as fracture initiator.

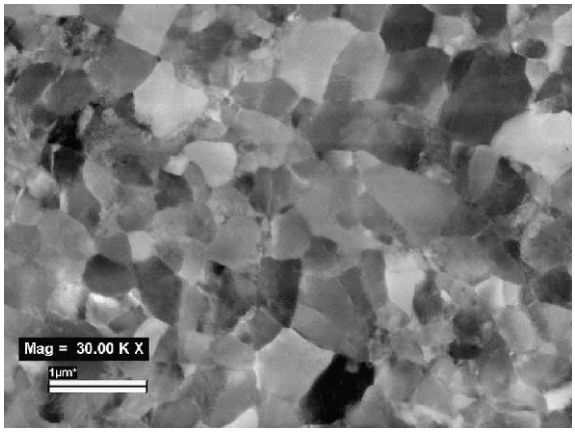

Fig. 4. Electron back scatter image of DP after HPT ($\phi \approx 108$).

The 'yield strengths' (plastic deflection of 0.01 mm) of the samples which have been deformed to higher deformation degrees ($\phi \approx 2.77$ and 5.55) are even about three times larger than the bending fracture strengths of DP-R. Again ductility has been observed at RT but since there has also crack nucleation during SPD taken place the amount of ductility is again limited.

Only the tested specimen with the deformation $\phi \approx 5.55$ shows a higher degree of ductility (bendingangle $>$ 30°, specimen width 6 mm, span width 22 mm). A possible explanation is that the formation of new small 'grains' increases the fracture toughness and hence the small cracks do not reduce the ductility during bending. To verify this assumption, K_{Ic} tests will be performed to determine the fracture toughness.

5. Conclusions

Pure chromium in the recrystallized condition behaves brittle at RT. Small pre-deformations above the DBTT result in ductility in bending and tension at RT.

SPD of pure chromium results in an increase of strength and ductility. However, the obtained ductility is limited due to the formation of small cracks during the SPD process.

After an applied strain of $\phi \approx 5.55$, the formation of new regions with a size smaller than 2 μm sets in, leading to an increase of fracture toughness and hence also an increase of ductility in bending and tensile experiments.

HPT tests indicate that a fine equiaxed microstructure develops during further severe plastic deformation. The evaluation of the microstructure with increasing deformation grade in chromium is similar to that observed in copper or aluminum [2,5].

The critical point for fusion application, the thermal stability of these microstructures will be investigated in our present work. Since the field of SPD is very young until now it is only possible to produce small scaled components but this limitation will be overcome in the next years.

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