ORAL REFERENCE: 100439

FRACTURE IN CHROMIUM: AN ATTEMPT TO IMPROVE DUCTILITY

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ABSTRACT

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 **R**oom **T**emperature (RT) and their high **D**uctile to **B**rittle **T**ransition **T**emperatures (DBTT). The investigated specimens (pure chromium with a purity of about 99.97%) have been produced in a powder metallurgical route. They have been tested in the as **HIP**ped (Hot Isostatic Pressed) condition and after different pre-deformations. The as HIPped microstructure shows in bending tests and tension tests brittle behavior at RT and a steep increase of ductility in the temperature range between 200°C and 250°C. Fracture toughness investigations reveal a DBTT between 290°C and 300°C. The K_Q value increases from 12 MPa√m at 290°C up to a value exceeding 500 MPa√m at 320°C. Additional to the static fracture toughness investigations also fatigue crack growth tests were performed at RT and above DBTT in order to improve the understanding of the fracture processes. An improvement in ductility and an increase in bending fracture strength have been induced by pre-deformation in tension, in bending, by single passed **E**qual **C**hannel **A**ngular **E**xtrusion (ECAE) and by die compression.

KEYWORDS

ECAE, fracture toughness, cleavage fracture, DBTT, fatigue, ductility, chromium

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 **R**oom **T**emperature (RT) and their high **D**uctile to **B**rittle **T**ransition **T**emperatures (DBTT). In this paper mechanical and fractographical investigations are presented of pure chromium (DUCROPUR) with a purity of about 99.97% (N: 19µg/g, C: 51µg/g, O: 40µg/g, H: 2µg/g). The specimens have been produced in a powder metallurgical route by the company Plansee. The density after **H**ot **I**sostatic **P**ressing (HIP) was 7.165g/cm^3 (99.51%) and the grain size was about 100µm. The material has been investigated in the as HIPped condition (recrystallized microstructure) and after different pre-deformations.

EXPERIMENTAL PROCEDURES

Different tests have been performed to characterize the mechanical properties of recrystallized pure chromium (DP-R). Notched and in compression pre-cracked 3-point bending samples (6x6x30mm) have been used to determine the fracture toughness below the DBTT. The span of the rolls was 24mm. Above this temperature J-tests using CT-specimens (W=25mm, B=12mm, a=12mm) have been carried out to measure the value of fracture toughness. To determine the DBTT and the strengths as a function of temperature of uncracked specimens 3-point bending tests (samples 6x6x30mm) and tension tests (samples ∅=3.5mm, l_0=17.5mm), have been performed. All samples have been mechanically polished and electropolished to minimize the surface influence. Furthermore some simple compression tests (samples 6x6x12mm) have been done to determine the yield strengths below the DBTT. To improve the understanding of the fracture processes fatigue crack growth tests (CT-specimens W=25mm, B=6mm, a=12mm) have been performed in air at RT and above DBTT.

Recrystallized (as hipped) tensile and bending specimens have been plastically deformed above the DBTT. These pre-deformed samples have been tested at RT. The crosshead velocity in the bending tests was 0.08mm/min and the strain rate in the tensile tests was $\dot{\varepsilon}=7.6\text{x}10^{-5}\text{s}^{-1}$.

Samples have been pre-deformed by single passed **E**qual **C**hannel **A**ngular **E**xtrusion (ECAE) [1] at 320°C and by die compression at RT. From these resulting samples small specimens have been machined using a diamond wire saw. Subsequently, they have been heat treated in air for 80 minutes at 300°, 500° and 700°C and tested in bending (ECAE: span of the rolls=16.5mm, die compression: span of the rolls=22.5mm) at RT.

RESULTS AND DISCUSSION

Tests on recrystallized samples

Fracture toughness investigations

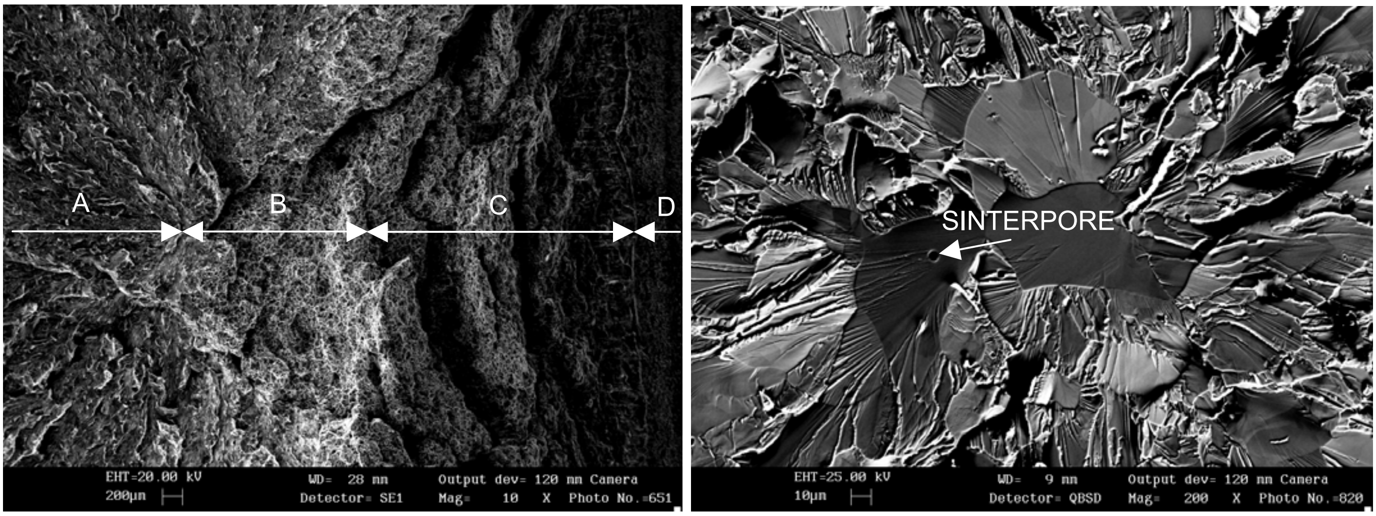
The obtained fracture toughness values of the performed K- and J-tests are summarized in Tab.1. The specimen size criterion of the ASTM standards was not fulfilled for the toughness values which are marked by a star.

TABLE 1

K_{IC}- OR K_Q-VALUES OF RECRYSTALLIZED PURE CHROMIUM (DP-R) IN MPA√M

T[°C]	DP-R
-196	3.0
25	7.7
100	11.7*
200	11.6*
280	11.8*
290	17.5*
320	~500*
480	500*-660*

From liquid nitrogen temperature (-196°C) up to 290°C the toughness values increase slightly with increasing temperature. No macroscopic plastic deformation has been observed. The fracture surfaces are characterized by cleavage planes. Above 300°C the fracture behavior changes rapidly and dramatically. The samples show large plastic deformations at the crack tip before fracture (CTOD≈1.4mm). The toughness values calculated from the J-tests are 500 MPa√m at 320°C and between 500 and 660 MPa√m at 485°C. The fracture surface of a sample tested at 485°C is depicted in Fig.1a. Similar to the fracture surface of the specimen tested at 320°C it shows an extensive crack tip blunting. The stable ductile crack propagation has been observed only at 485°C. In spite the large toughness values the final fracture occurs again in a cleavage mode.



(a) b)

Figure 1: Fracture surfaces of (a) a J-integral sample tested at 485°C (A...final cleavage fracture, B...stable ductile crack growth, C...blunting zone, D...fatigue pre-crack) and (b) a bending sample tested at RT

Bending tests

The obtained results are summarized in Fig. 2. Within the DBTT range (which is here somewhat lower than in the fracture toughness tests) some samples fail in a brittle mode and some show large plastic deflections. Below 200°C no large plastic deflections have been observed. Above DBTT the test has been interrupted after a plastic deflection of about 2mm without final fracture.

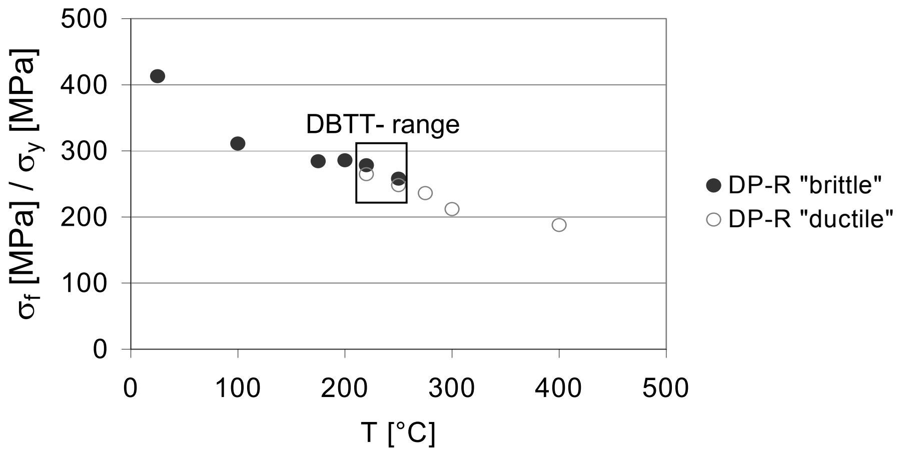


Figure 2: Dependence of bending strengths on temperature; the full symbols indicate fracture strengths (σ_F), the open symbols indicate "yield strengths" (σ_y) at the plastic deflection of 0.01mm

At RT and at a temperature somewhat smaller than DBTT a Weibull characterization has been performed. The Weibull modulus increases from m=20.7 at RT up to m=59.2 at 175°C. In both tests series the initiation points of fracture are usually sinterpores (Fig.1b).

Tension tests

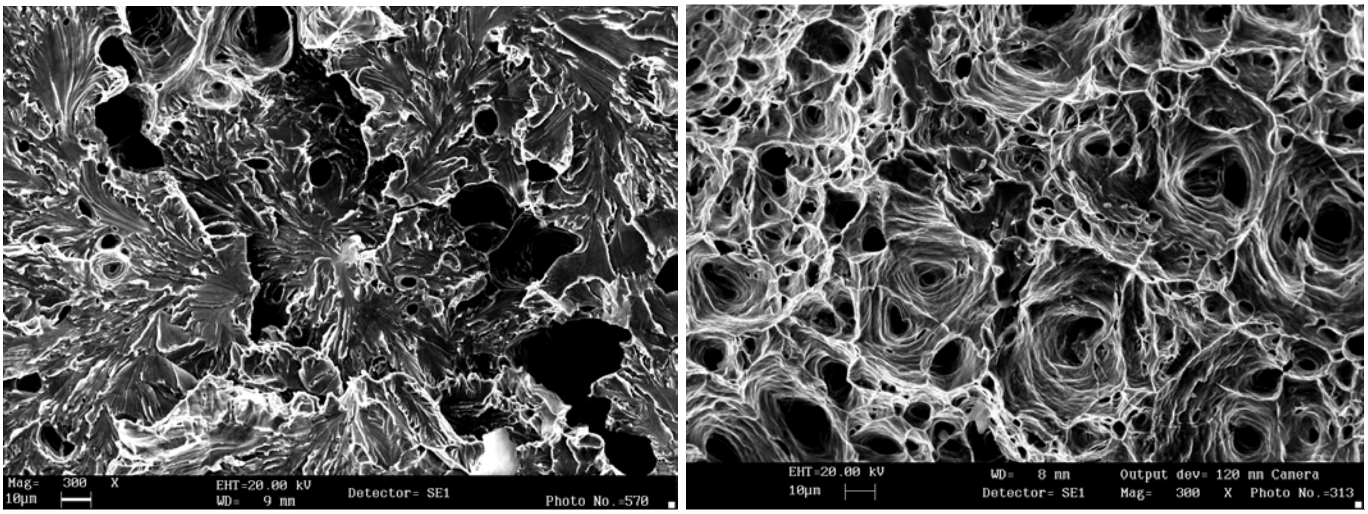
The obtained strengths in MPa and strains in % are listed in Tab. 2. $\sigma_{0.2}$ is the 0.2% offset yield strength, σ_{UYP} the upper yield point and σ_{UTS} the ultimate tensile strength. ε_a is the transversal strain and ε_l the longitudinal strain which is related to 5 times the sample diameter.

TABLE 2

TENSILE STRENGTHS IN MPA AND STRAINS IN %

T [°C]	$\sigma_{0.2}$ or σ_{UYP}	σ_{UTS}	ε_a	ε_l
25	319.0	319.0		0.6
200	*199.3*	195.8	2.9	3.1
300	*121.1*	244.8	79.8	52.6
400	108.5	249.8	84.7	49.4
600	113.1	219.05	87.9	36.9
800	82.6	151.9	95.3	38.3
960	63.1	85.3	84.7	29.7

The fracture surfaces of DP-R below DBTT are again characterized by cleavage planes. Somewhat above the DBTT large plastic deformations take place but beside void formation also cleavage planes are observed on the fracture surface (Fig.3a). With increasing temperature the cleavage planes disappear and the void formation increases (Fig.3b).



(a) (b)

Figure 3: Fracture surfaces of DP-R tensile specimens (a) tested at 300°C and (b) tested at 400°C

Compression tests

The upper yield point is 402.2MPa and 183.3MPa at RT and 170°C, respectively. At RT the upper yield point is about 25% larger than the fracture stress in tension. In contrast to the tension tests large plastic deformations can be obtained without failure.

Fatigue crack growth tests

Fatigue crack growth experiments were performed at RT and somewhat above DBTT (300°C). The stepwise increasing load amplitude test [2] on compression pre-cracked specimens was applied, which allows to measure fatigue crack growth behavior between the effective threshold and the long crack threshold. At RT the long crack threshold of stress intensity range (at a stress ratio of 0.1) and fracture toughness are approximately the same. However below the long crack threshold the crack propagates over short distances where the fracture surface showed features of a typical ductile metal. This indicates that a certain plastic deformation of the crack tip is possible also at RT. At 300°C a fatigue crack growth behavior typical for ductile metals was observed. An effective threshold $\Delta K_{eff,th} \approx 3.5 MPa\sqrt{m}$ and a long crack threshold $\Delta K_{th} \approx 7.5 MPa\sqrt{m}$ have been determined at a stress ratio of 0.1.

Tests on pre-deformed samples

It had been reported [3-5] that chromium can be ductile at RT. Beside the amount of the interstitial solved impurities (N, C,...) the dislocation density plays an important role. Recrystallized chromium has been found usually brittle at RT whereas pre-deformed samples showed sometimes ductility at RT. Therefore samples have been pre-deformed to investigate the influence of dislocation density on ductility at RT.

Pre-deformation in bending and tension

Bending samples of DP-R have been plastically pre-deformed at 400°C (deflection about 1mm). After that the samples have been tested at RT. In contrast to the tests with DP-R the pre-deformed samples show ductility at RT. A plastic deflection of about 2 mm is obtained before the final fracture occurs in a brittle mode.

Similar to the pre-deformation in bending tension samples of DP-R have been pre-deformed (ε_{pl}= 3%, 5% or 10%) at about 300°C. The stress-strain curve indicates a pronounced yield point and an area of "Lüders strain". This assigns a not negligible 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.

Pre-deformation by ECAE

A 15x15x30mm block was deformed by ECAE at a temperature of about 320°C. The channel angle was 120° which corresponds to a true strain of $\phi \approx 1$. After a single passed deformation the samples were heat treated. The obtained fracture strengths σ_F and fracture bending angles α_F are listed in Tab.3. Samples without heat treatment and after heat treatments up to 500°C show again a limited ductile behavior. The initiation points of fracture are mostly sinter defects (Fig.4a). The specimens after a heat treatment at 700°C fail in a completely brittle mode again. All bending fracture strengths are about 2 times larger than those of DP-R. An attempt to improve the ductility will be a multiple passed ECAE above the DBTT (work in progress). The aim is to obtain a finer grain size which is not the case by single passed ECAE.

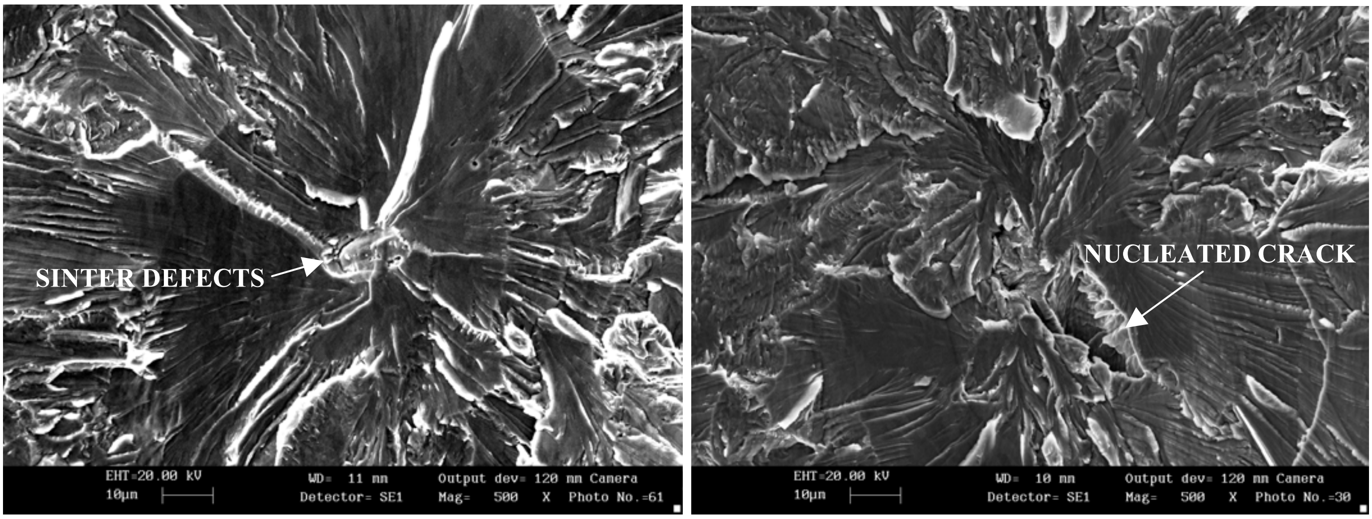
TABLE 3

BENDING STRENGTHS (σ_F) IN MPA AND FRACTURE BENDING ANGLES (α_F) AFTER DIFFERENT HEAT TREATMENTS

	ECAE		DIE COMPRESSION	
Heat treatment after deformation	**σ_F [MPa]**	**α_F [°]**	**σ_F [MPa]**	**α_F [°]**
none	768.8	0.4	794.7	0.3
none	1031.3	1.9	761.6	0.2
80 minutes at 300°C	854.4	0.6	878.6	0.2
80 minutes at 300°C	932.1	1.0		
80 minutes at 500°C	899.1	0.3	743.7	
80 minutes at 500°C	1053.8	1.2	889.0	1.0
80 minutes at 700°C	727.9	0	641.3	0
80 minutes at 700°C	721.5	0		

Pre-deformation by die compression

A 15x15x30mm block was deformed by die compression at RT (true strain $\phi \approx 1$) and again heat treated. The obtained fracture strengths σ_F and fracture bending angles α_F are listed in Tab.3, too. Again only the samples after the heat treatment at 700°C show completely brittle fracture behavior. The bending fracture strengths and fracture bending angles are smaller than those of the tested samples after a pre-deformation by ECAE. Pre-deforming by die compression at RT have nucleated cracks, these cracks act as fracture initiator (Fig.4b) and hence no large plastic deflections are possible. To avoid this crack nucleation die compression should be applied above the DBTT (this work is in progress, too).



(a) (b)

Figure 4: Fracture point of initiation after pre-deformation by (a) ECAE and (b) die compression

CONCLUSIONS

- The investigated pure chromium in its recrystallized condition shows a rapid increase in fracture toughness between 290°C (17.5MPa√m) and 320°C (500 MPa√m). In the latter case the final fracture occurs after a large crack tip blunting in a brittle mode. Bending tests of smooth specimens show no plasticity at RT, above 220°C large plastic deformations are obtained. Fatigue crack growth tests reveal indications of ductility at RT and at 300°C the typical behavior of a ductile metal.
- A limited ductility in bending and an increase of the bending fracture strength results after different pre-deformations. The best results are obtained by small pre-deformations in bending. The pre-deformation by die compression at RT is characterized by nucleation of small cracks. Therefore no larger plastic deformations can be obtained.

ACKNOWLEDGEMENTS

This work has been carried out within Association EURATOM-ÖAW and is supported by the Bundesministerium für Wissenschaft, Forschung und Kunst.

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