

FRACTURE TOUGHNESS OF TWO Cr₂Hf+Cr INTERMETALLIC COMPOSITES AS A FUNCTION TEMPERATURE

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ABSTRACT

Fracture toughness as a function of temperature was evaluated for two Cr₂Hf+Cr intermetallic composites, each in two different microstructural conditions. The proeutectic microstructures based on Cr-6.5Hf (at%) showed a significant increase in fracture toughness with an increase from room temperature to 600°C. The coarse microstructure obtained by heat treatment at 1500°C showed evidence of ductile behavior of Cr at a lower test temperature (200°C) relative to that of one heat treated at 1250°C (400°C). In the eutectic microstructures based on Cr-13Hf, only a small increase in fracture toughness at 600°C was seen. The results are analyzed in the light of fracture micromechanisms.

INTRODUCTION

Fracture resistance is one of the primary requirements for successful implementation of intermetallics in high temperature structural applications. Fracture toughness of many intermetallics are low, often in the range of 1-5 MPa√m. The approach to increasing the fracture toughness through ductile particles is promising in a number of intermetallic systems, such as TiAl+Nb [1], Nb₅Si₃+Nb [2], MoSi₂+Nb/Ta [3] NiAl+Mo [4]. However, the requirements for high temperature use also include the thermochemical stability of phases or constituents at those temperatures. This has been a major concern in systems which are not in thermodynamic equilibrium, made by powder metallurgical techniques, since considerable reaction could occur at high temperatures causing interface reactions and loss of ductility of the toughening phase.

In-situ intermetallic composites, by virtue of their thermochemical equilibrium of phases show potential for high temperature applications [5]. Recent interest on systems such as Cr₂Hf+Cr [6,7], Cr₂Ta+Cr [6], Cr₂Nb+Cr [5] and Cr₃Si+Cr [8] is primarily based upon the fact that Cr is oxidation resistant up to 1000°C. The kinetics of oxidation of Cr is low relative to other refractory metals [9]. Cr also exhibits little weight gain up to about 1000°C [10].

In this investigation, fracture toughness levels of composites based on the Cr₂Hf+Cr system [11] were evaluated. In earlier works [6, 7], fracture toughness of a composite having a bulk composition of Cr-6.5Hf (at.%) was evaluated. In the present study, two composites having compositions of Cr-6.5Hf (proeutectic) and Cr-13Hf (eutectic), with two different microstructural conditions obtained by heat treatment, were selected for fracture toughness measurement. The objective is to evaluate fracture toughness as a function of temperature under both microstructures. Fractographic analyses were performed to determine fracture modes.

EXPERIMENTAL PROCEDURE

Alloy ingots with nominal compositions of Cr-6.5Hf (at%) and Cr-13Hf were arc melted and cast in copper molds. The ingots were enclosed in Mo cans and extruded at 1500°C with an approximate reduction ratio of 4:1. The extruded bars were decanned by electric discharge machining (EDM), and bars of size, 32mm X 6.35mm X 6.35mm were machined for fracture toughness testing. The bars were heat treated at 1250°C and 1500°C for 100 hrs. under the flow of high purity (>99.5%) gettered argon at a low rate and furnace cooled. Specimens were wrapped in Ta foils during heat treatment. Cr losses due to high vapor pressure at these temperatures were minimal. After heat treatment, specimen surfaces were polished to a 600 grit finish to remove the surface layer. Subsequently, notches of 2.5 mm in depth and about 50 μm in root radius were machined by EDM. Fracture toughness tests were performed at a displacement rate of 8.5×10^{-3} mm/min. in 3 point bending at room temperature and at 200, 400 and 600°C in argon atmosphere. Fracture surfaces were examined in a scanning electron microscope (SEM) to document fracture micromechanisms.

RESULTS AND DISCUSSION

The chemical composition of the alloys are presented in Table I. Microstructures in the as-extruded condition consisted of elongated primary Cr grains and eutectic structure in Cr-6.5Hf and a deformed eutectic lamellar structure in Cr-13Hf. Preliminary observations indicated that the microstructures recrystallized but did not coarsen up to 1300°C when heat treated for 100 hrs. Above 1300°C, microstructural coarsening was noticeable. Figs. 1 & 2 show the microstructures, as seen in longitudinal sections, after heat treatment at 1250°C and 1500°C for Cr-6.5Hf and Cr-13Hf alloy, respectively. Coarsening of the eutectic structure in both the alloys can be seen. Quantitative image analysis indicated that the relative volume fractions of Cr and Cr₂Hf differed only by a few % between the heat treatments in both the alloys.

Table I Chemical composition of alloys (wt.%)

Alloy	Hf	Zr	Cu	Fe	Ti	O	N	C
Cr-6.5Hf	17.1	0.15	0.02	0.08	0.03	0.044	0.005	0.0076
Cr-13Hf	33.1	0.2	0.04	0.07	0.02	0.032	0.005	0.0067

Results of fracture toughness tests are presented as a function of temperature in Figs. 3a&b for the heat treatment and alloy combinations studied. In both fine and coarse microstructural conditions, the proeutectic alloy shows an increase in fracture toughness (K_{Ic}) from about 7 MPa $\sqrt{\text{m}}$ at room temperature to 15-18 MPa $\sqrt{\text{m}}$ at 600°C. Fractographic examination revealed a cleavage fracture mode (Figs. 4a&b) suggesting that both the Cr and Cr₂Hf phases are brittle at room temperature. At 600°C, Cr exhibited ductility and Cr₂Hf failed in a brittle manner. Ductile rupture of the Cr phase surrounded by Cr₂Hf cleavage regions can be seen in Figs. 4c&d. At 200°C, fracture toughness of the coarse microstructure is significantly higher compared to that of the fine microstructure (13 vs. 9 MPa $\sqrt{\text{m}}$). While Cr grains which fractured in a ductile fashion could be rarely seen in the fine microstructure, several Cr grains exhibiting ductile fracture and debonding were seen in the coarse microstructure (Figs. 4e&f).

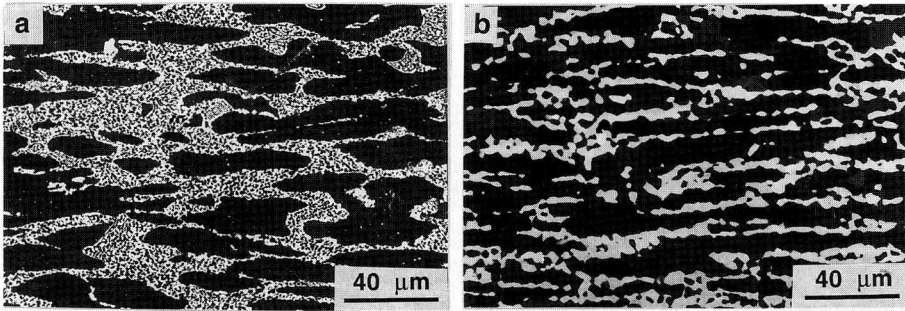


Fig. 1. Microstructures of Cr-6.5Hf alloy after heat treatment at (a) 1250°C and (b) 1500°C for 100 hrs.

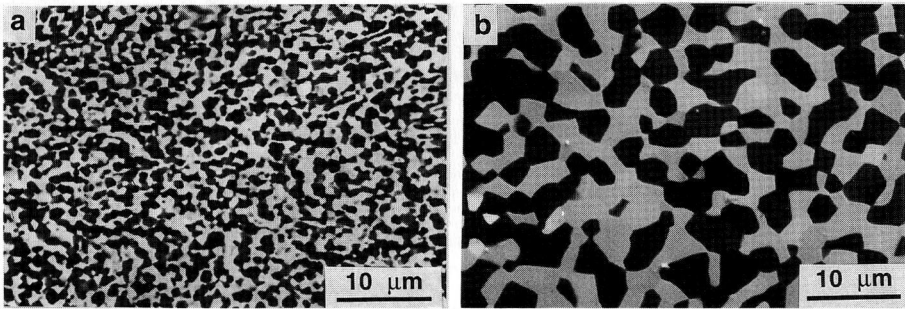


Fig. 2. Microstructures of Cr-13Hf alloy after heat treatment at (a) 1250°C and (b) 1500°C for 100 hrs.

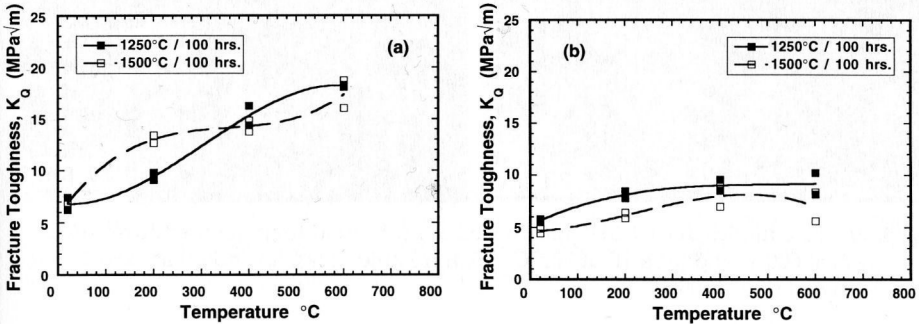


Fig. 3. Fracture toughness as a function of temperature for (a) Cr-6.5Hf and (b) Cr-13Hf alloy.

Fracture toughness levels in both the fine and the coarse eutectic microstructures increase only slightly with an increase in test temperature (Fig. 3c). At all temperatures, the coarse microstructure exhibited lower K_{Ic} (by about 1-2 $\text{MPa}\sqrt{\text{m}}$) values relative to the fine microstructure. Similar to the proeutectic alloy, the fracture mode was completely brittle in RT tests (Fig. 5a&b) and partially ductile due to the plastic

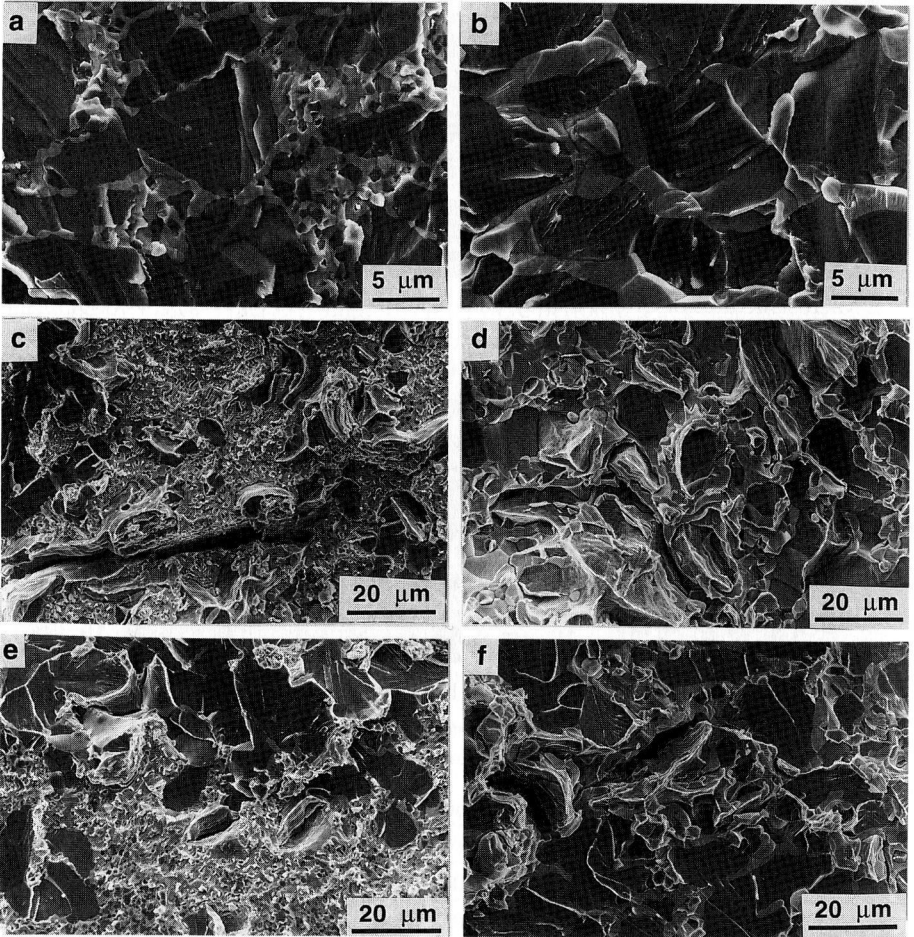


Fig. 4. Fracture modes in Cr-6.5Hf alloy tested at (a) & (b) at room temperature ; (c) & (d) at 600°C and (e) & (f) at 200°C. Heat treatment: (a), (c) and (e) at 1250°C; (b), (d) and (f) at 1500°C.

deformation of Cr at 600°C (Figs. 5c&d). There was no discernible difference in fracture mechanism between coarse and fine eutectic microstructures at all temperatures.

In all microstructures, it is evident that the transition from brittle to ductile failure of Cr results in the increase in fracture toughness with temperature. It is well known [12, 13] that the brittle to ductile transition temperature (BDTT) of Cr depend on prior deformation history, interstitial content (O, N and C), presence of elements in solid solution, surface finish etc. In these investigations, the BDTT temperature was

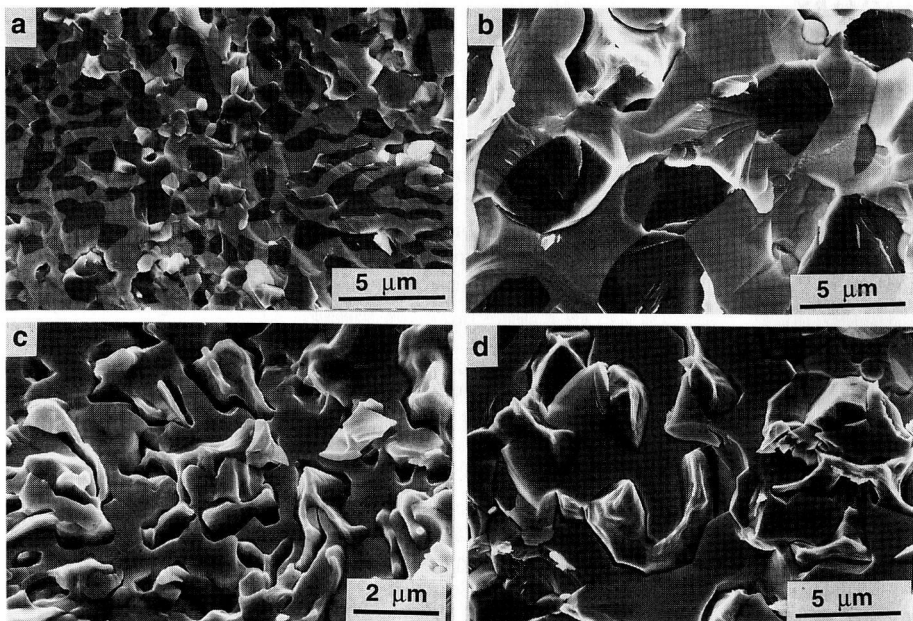


Fig. 5. Fracture modes in Cr-13Hf alloy tested at (a) & (b) at room temperature ; (c) & (d) at 600°C. Heat treatment: (a) and (c) at 1250°C; (b) and (d) at 1500°C.

found to vary from $<0^{\circ}\text{C}$ to 800°C depending on the above variables. In particular, interstitial content has been known to have a strong effect on the BDTT. Although the N content in the present alloys are lower than the reported [12, 13] levels required to cause brittleness in Cr, O levels are higher, falling in the range of compositions causing brittle failure in Cr.

A noticeable feature in the microstructures of the proeutectic alloy is that after heat treatment at 1250°C , fine particles, much smaller in size compared to Cr_2Hf phase, were seen at and in the interior of recrystallized primary Cr grains. These are absent after heat treatment at 1500°C . Internal precipitation in Cr, yet unidentified, were seen in thin foils under TEM for both heat treatment conditions. Further work is in progress to identify the factors causing the different Cr behavior (brittle vs. ductile), which resulted in the high K_{Ic} values at 200°C for the microstructure heat treated at 1500°C .

The reasons for the decrease in fracture toughness in the eutectic microstructure after coarsening are not clear. The volume fractions of Cr and Cr_2Hf in both heat treatments were nearly the same and the phases are co-continuous. According to models on ductile phase toughening [14, 15], the coarse microstructure with a Cr particle size of about 4 times that in the fine microstructure should result in an increase in toughness at least by a factor of 2. It should be noted that the size of the Cr_2Hf also is higher by the same amount, and could lead to a larger perturbation of crack front in Cr_2Hf . It appears that the reduction in K_{Ic} due to increased size of brittle fracture of Cr_2Hf outweighs the increase in toughness due to increased ductile particle size.

SUMMARY

Due to the brittle behavior of Cr, fracture toughness levels at room temperature were only modest (5-7 MPa√m) in both Cr-6.5Hf and Cr-13Hf alloys with proeutectic and eutectic microstructures respectively. However, Cr showed extensive ductility at 600°C and resulted in a significant increase in fracture toughness to 15-18 MPa√m in Cr-6.5Hf. In Cr-13Hf alloy with eutectic microstructure an increase in temperature caused only a small increase (1-2 MPa√m) in fracture toughness, possibly due to the opposite effects of ductile Cr phase and brittle Cr₂Hf phase on crack resistance. It appears that minimization of microstructural continuity of Cr₂Hf, in addition to an increase in the amount and size of ductile particles, could increase fracture toughness.

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