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PROCESSING AND PROPERTIES OF EXTRUDED TUNGSTEN-HAFNIUM AND TUNGSTEN-STEEL COMPOSITES

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ABSTRACT

The purpose of this study was to evaluate the processing behavior and properties of tungsten-hafnium (W-Hf) and W-steel composites produced by hot extrusion of canned powders. The W-Hf composite was consolidated by extrusion of blended powders with preheat temperatures over the temperature range of 1100 to 1400°C. All extrusions produced fully dense material which exhibits elongation of the tungsten phase within the hafnium matrix. The flow stress, as characterized by the extrusion constant, decreases with increasing temperature up to 1300°C and increases substantially at 1400°C as significant quantities of intermetallic phase are formed during preheating. The room-temperature (RT) hardness and compressive yield stress increase modestly with increased extrusion ratio and are not affected by extrusion temperature in the range 1100 to 1300°C. The microstructures are essentially fully recrystallized at the 1300°C preheat temperature and partially recrystallized at lower temperatures. Additionally, a mixture of tungsten and steel powder was consolidated to full density by hot extrusion at a 1000°C preheat temperature and a reduction ratio of 4.2. Increased reduction of the W-steel composite results in increased RT hardness.

INTRODUCTION

The purpose of this study was to evaluate the processing behavior and properties of tungsten-hafnium (W-Hf) and W-steel composite materials produced by hot extrusion of canned powders. This technique is of interest because it is applicable to a wide variety of matrix materials and typically produces fully dense material over a broad range of process parameters. The extrusion preheat temperatures can frequently be maintained sufficiently low to avoid unwanted interactions between the tungsten phase and the matrix material.

PROCEDURE

The starting materials for the W-Hf composite were tungsten powder with an average particle size of 70 µm and hafnium powder with an average particle size of about 50 µm. The starting materials for the W-steel composite were tungsten powder with an average particle size of 12 to 15 µm and steel powder with an average particle size of 10 µm. The tap density of the powders was about 50% of theoretical density. The powder characteristics and preparation of canned powder for extrusion have been previously described [1].

The canned W-steel powders were heated in air for 1 h at 1000°C. At higher preheat temperature, an argon cover was used to minimize oxidation of the can exterior. Transfer time to the extrusion press from the furnace is typically 15 s, and total time of extrusion is typically 30 s. The load and ram positions were recorded as a function of time during the extrusion. Extrusion ratios of 4.2 to 9.0 were employed for the W-Hf composite. In the case of the W-steel material, extrusion ratios above 4.2 resulted in force requirements exceeding those available, and only hot-pressed materials were obtained. A re-extrusion of each material was performed by cutting an extruded section to about 15-cm length and placing it in a cavity of slightly larger dimensions within another can of the same material.

The extruded materials were examined by optical metallography. The W-steel material was photographed in the as-polished condition. The W-Hf material was photographed after being etched in a solution of 98% concentrated nitric acid and 2% concentrated hydrofluoric acid. Samples of both materials were also etched in a boiling solution of one part concentrated hydrogen peroxide and three parts water in order to reveal the structure of the tungsten phase.

Hardness testing was performed using a Vickers indenter at a load of 500 g. Compression testing was conducted at room temperature (RT) on samples of 7.6 mm diam and 15.2 mm length at a rate of 10^{-3} s^{-1}.

RESULTS

The extrusion parameters used for the W-Hf composites are shown in Table 1 with the measured maximum extrusion force. An extrusion constant, K, may be obtained from the equation for the force, P, as:

$$P = K A_0 \ln (A_0/A_f) , \qquad (1)$$

where

A_0 = area of opening of the extrusion liner,
A_f = final area of the extruded cross section.

Table 1. Summary of extrusion data for canned tungsten-hafnium powder

Preheat temperature (°C)	Can material	Extrusion diameter (mm)	Ratio	Maximum load (MN)	Extrusion constant (MPa)
1100	316 Stainless	21.1	6.1	3.6	825
1200	316 Stainless	21.1	6.1	3.5	805
1300	Molybdenum	25.4	4.2	1.5	435
1300	Molybdenum	20.7	6.4	1.8	405
1300	Molybdenum	17.4	9.0	2.2	415
1400	Molybdenum	21.1	6.1	4.0	915

The extrusion constant calculation is based on the assumption that the flow stress is proportional to the true strain and, thus, ignores frictional effects as well as other details of the deformation process. It is dependent on material, temperature, and strain rate but is still of use in comparing and predicting extrusion loads. The extrusion constant is plotted as a function of preheat temperature in Figure 1. The extrusion constant for the W-Hf composite decreases with increased preheat temperature up to 1300°C and then increases in association with intermetallic formation.

The extrusion loads for the three extrusions of W-steel with a 1000°C preheat temperature and 4.2 ratio varied from 2.8 to 3.2 MN, corresponding to extrusion constants of 810 to 940 MPa. All extrusions exhibited a uniform cross section along the length.

The microstructure of the longitudinal and transverse sections of the W-Hf powder extruded with a 1300°C preheat temperature is shown in Figures 2 and 3 for the extrusion ratio of 4.2. The higher the extrusion ratio, the more elongated are the tungsten and hafnium regions in the longitudinal sections. The transverse sections show a progression with increasing extrusion ratio from more or less equiaxed polygonal tungsten regions to increasingly distorted regions. As seen in Figure 4, at higher magnification, the hafnium has been recrystallized during the extrusion to produce a grain size of 2 to 5 μm. The tungsten grain size shown in Figure 5 is also in the range of 2 to 5 μm. There is evidence of cold work in the tungsten phase, especially in regions which appear to be deformed by contact of adjacent tungsten particles without separating matrix phase. The grain size is similar over the range of extrusion temperatures investigated.

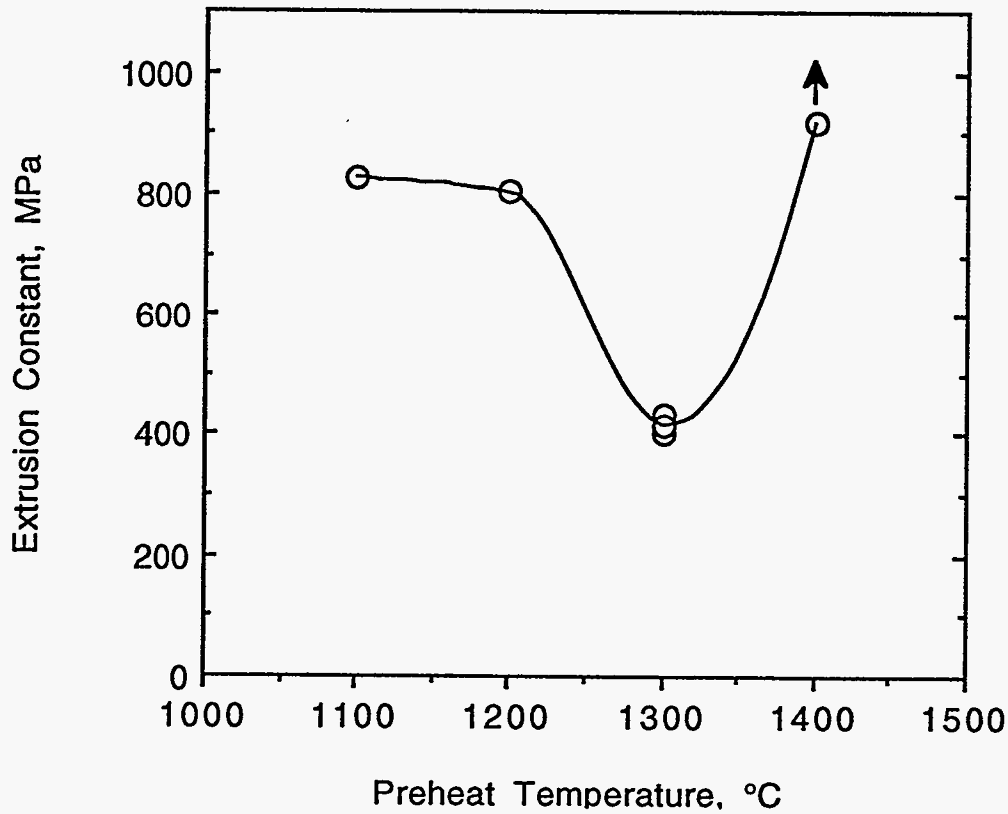

Figure 1. Extrusion constant for tungsten-hafnium powder decreases with increased preheat temperature up to 1300°C and then increases in association with intermetallic formation.

The size and shape of the phases obtained at the preheat temperatures of 1100 and 1200°C are essentially the same as those at 1300°C. However, there are large regions of unrecrystallized material as seen in Figure 6. At the 1400°C preheat temperature, a large amount of intermetallic phase is present at the boundaries of the tungsten phase with the hafnium phases, as seen in Figure 7. The intermetallic phase is expected to be HfW_2, which is the only stable intermetallic phase [2]. The intermetallic phase is formed during preheating of the powder. Its resistance to deformation is the likely cause of the rapid increase in the extrusion constant as the preheat temperature increases above 1300°C.

The microstructures of the hot-compacted and extruded W-steel powders are similar to those seen for the W-Hf composites. The hot-compacted material resulting from an unsuccessful extrusion shows only a small amount of residual porosity, on the order of 2%. Full density is

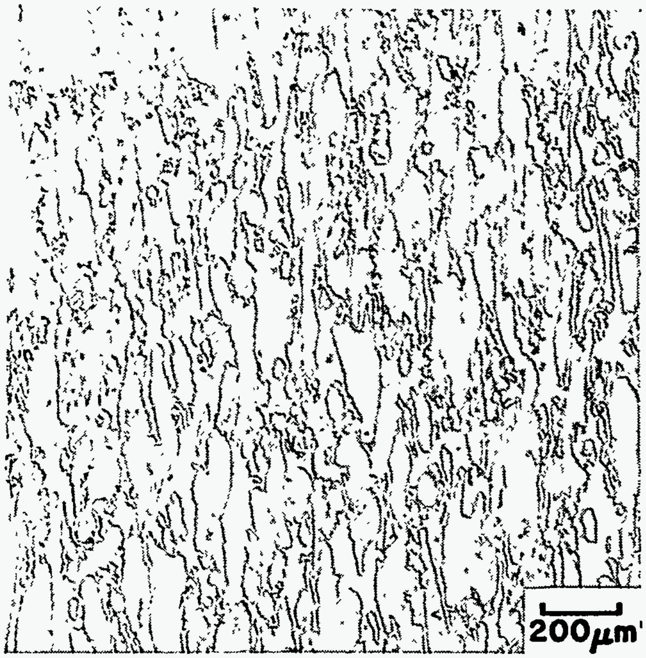

Figure 2. Longitudinal section of tungsten-hafnium composite extruded at 1300°C at a ratio of 6.4.

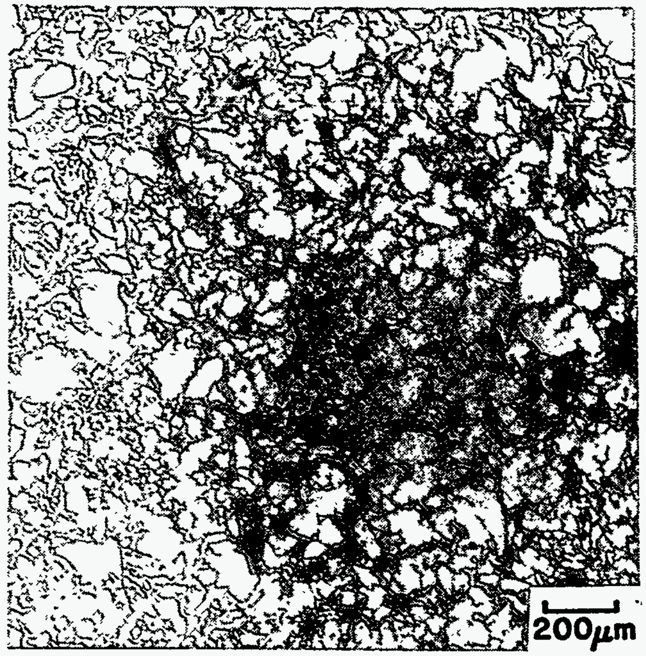

Figure 3. Transverse section of tungsten-hafnium composite extruded at 1300°C at a ratio of 6.4.

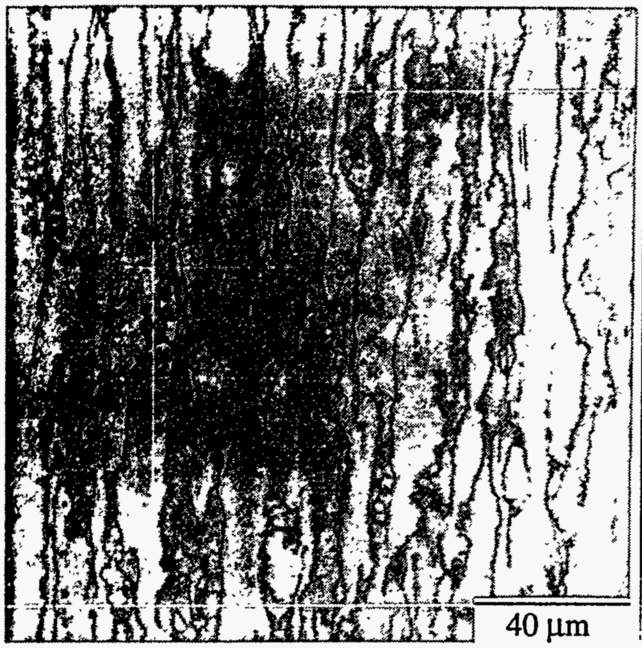

Figure 4. Tungsten-hafnium composite extruded at 1300°C shows recrystallized hafnium matrix.

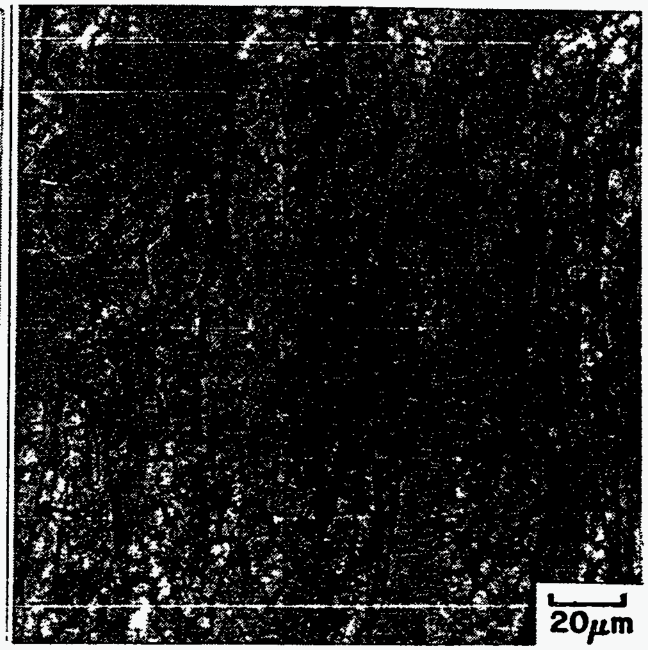

Figure 5. Tungsten-hafnium composite extruded at 1300°C shows equiaxed tungsten grains with localized deformation.

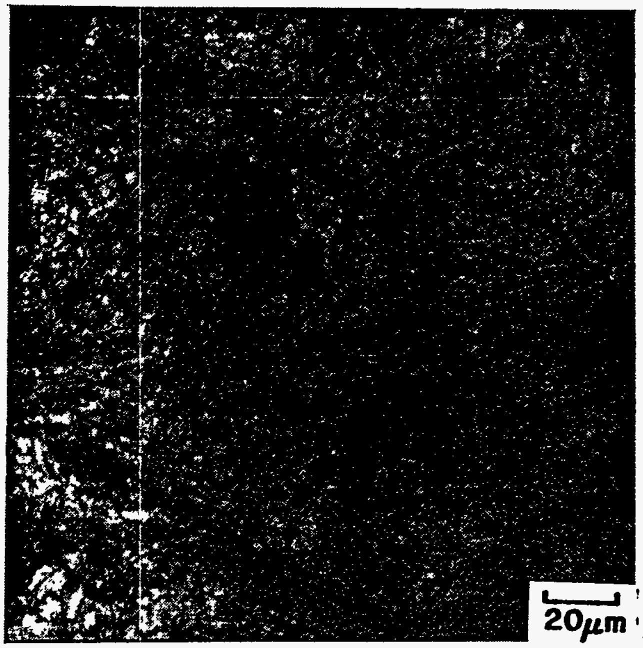

Figure 6. Transverse section of tungsten-hafnium composite extruded at 1200°C at a ratio of 6.1 and etched to reveal tungsten grain structure.

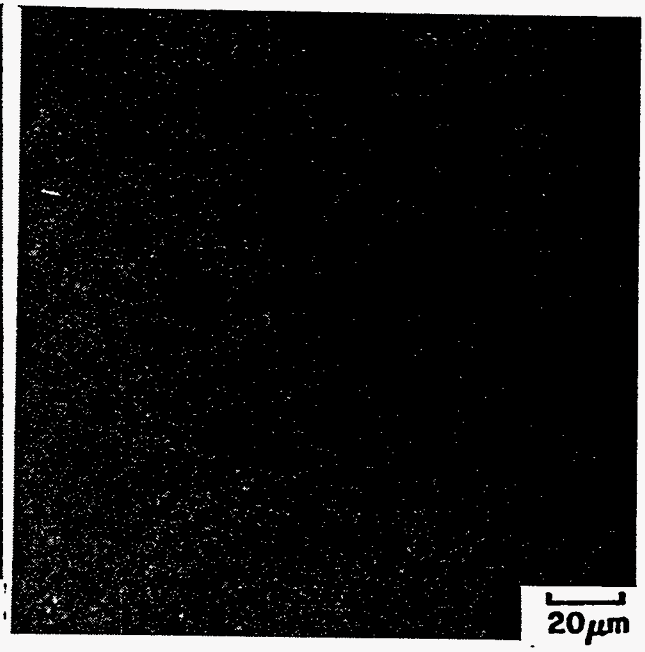

Figure 7. Tungsten-hafnium composite extruded at 1400°C shows intermetallic layer at phase boundaries.

achieved at an extrusion ratio of 4.2 or higher. The apparent tungsten grain size of 5 to 10 μm is similar to that in the W-Hf composite. The appearance of both equiaxed and deformed tungsten grains is also similar to that of the W-Hf composite, even for the extreme case of the re-extruded W-steel powder. There is some evidence of reaction between the tungsten and the steel.

The microhardness of the composites is shown in Figure 8 as a function of extrusion ratio for W-steel composites extruded at 1000°C preheat temperature and for W-Hf composites extruded at 1300°C preheat temperature. The hardness increases with increased extrusion ratio for W-steel composites and is independent of extrusion ratio for W-Hf composites.

The compressive yield and ultimate strengths are plotted versus extrusion ratio in Figure 9. The ultimate strength in compression decreases with increased extrusion ratio. The compressive yield shows a slight increase with increased extrusion ratio. The strain at fracture shows no trend and is in the range of 4.2 to 5.6% for the W-Hf composite. The W-steel composite has a yield strength of 1270 MPa and an ultimate strength of 1510 MPa in compression with a strain at fracture of 3.2%.

The fractograph of the compression test sample of the W-Hf composite extruded at 1200°C is shown in Figure 10. The fracture is by cleavage in the large tungsten grains and by intergranular separation in the fine recrystallized hafnium matrix phase. The fracture of the compression test sample of the W-steel composite, seen in Figure 11, shows cleavage of the tungsten and shear failure of the steel matrix. The cleavage surfaces of the tungsten grains are also smaller, as is expected from the smaller grain size of the tungsten powder and the absence of recrystallization and grain growth.

DISCUSSION

The results of the microstructural analysis indicate that the extrusion of W-Hf composite at a temperature of 1300°C results in a nearly fully recrystallized structure, while extrusion at 1100 or 1200°C results in only partial recrystallization of both tungsten and hafnium phases. This result is

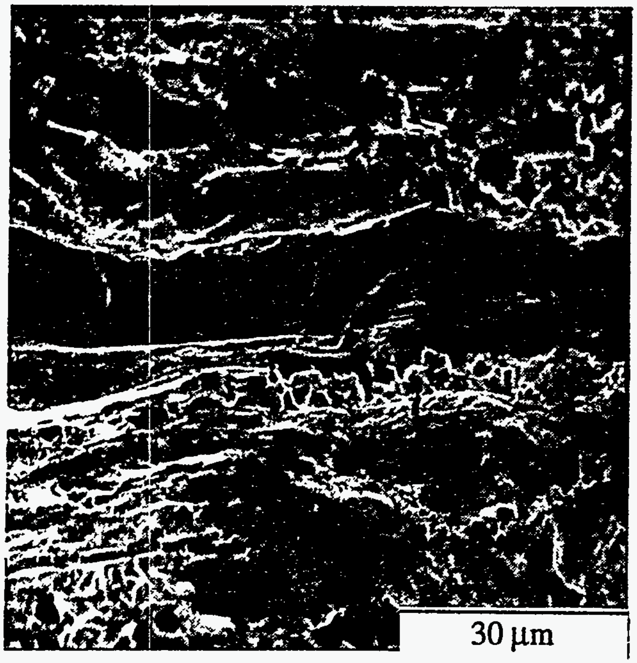

Figure 10. Fractograph of tungsten-hafnium composite extruded at 1200°C shows cleavage fracture of tungsten grains and grain boundary separation of hafnium.

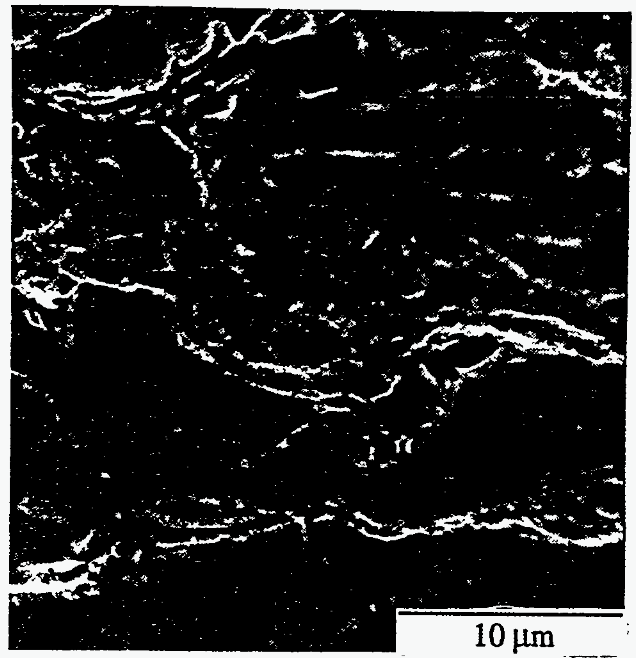

Figure 11. Fractograph of tungsten-steel composite extruded at 1000°C shows cleavage of tungsten grains and shear of steel matrix.

also consistent with a measured extrusion constant at 1300°C, which is only about half of the value measured at the lower temperatures. The fact that the RT microhardness and compressive yield stress of the W-Hf composite extruded at 1300°C do not change over a large range of extrusion ratios is also consistent with the recrystallization of the material during extrusion.

The extrusion of the W-steel composite produces, at most, a partial recrystallization of the tungsten phase. Since the initial crystallite size is similar to that in the extruded product, it is possible that the regions of equiaxed grains are undeformed rather than recrystallized. The absence or limited amount of recrystallization is consistent with the observed increase in microhardness with increased extrusion ratio.

The observed recrystallization behavior is in reasonable agreement with that of pure tungsten. The recrystallization temperature of tungsten depends on impurities and amount of work and is in the range of 1200 to 1400°C for commercially pure tungsten for times on the order of 1 h [ref. 3].

The morphology of the tungsten region, especially in the transverse section; is unusual. The highly irregular phase boundary, which becomes increasingly irregular with additional reduction, appears consistent with localized intergranular cracking of the tungsten and deformation of the continuous phase into the crack or separation.

CONCLUSIONS

The following conclusions can made from this study of consolidation of W-Hf and W-steel composites by hot extrusion of canned powders:

1. A W-Hf composite can be consolidated by hot extrusion to full density at preheat temperatures of 1100 to 1300°C with minimal material reactions. At 1400°C, intermetallic formation leads to increased extrusion loads.

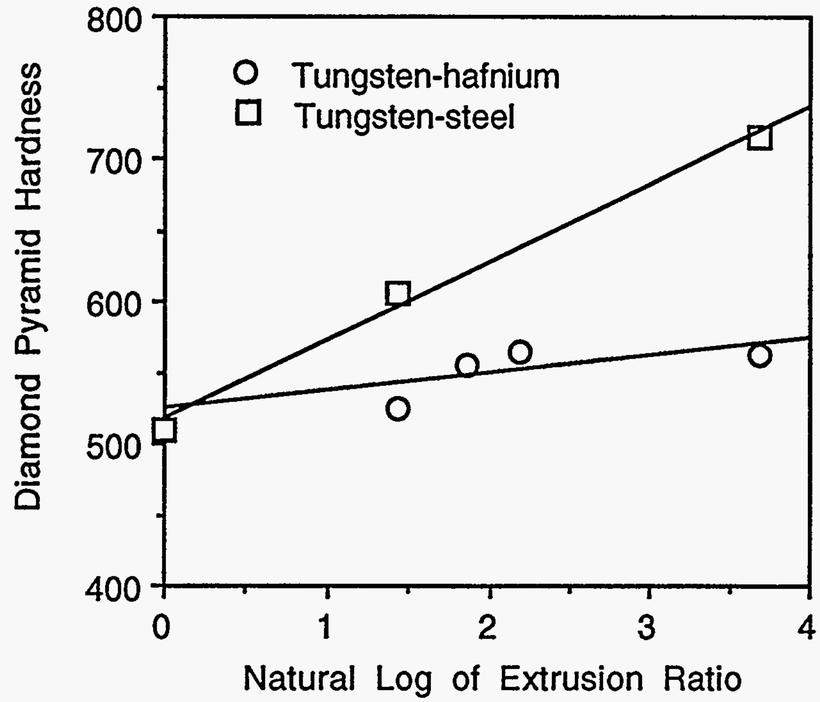

Figure 8. Hardness increases with increased extrusion ratio for tungsten-steel composites extruded at 1000°C preheat temperature and is independent of extrusion ratio for tungsten-hafnium composites extruded at 1300°C preheat temperature.

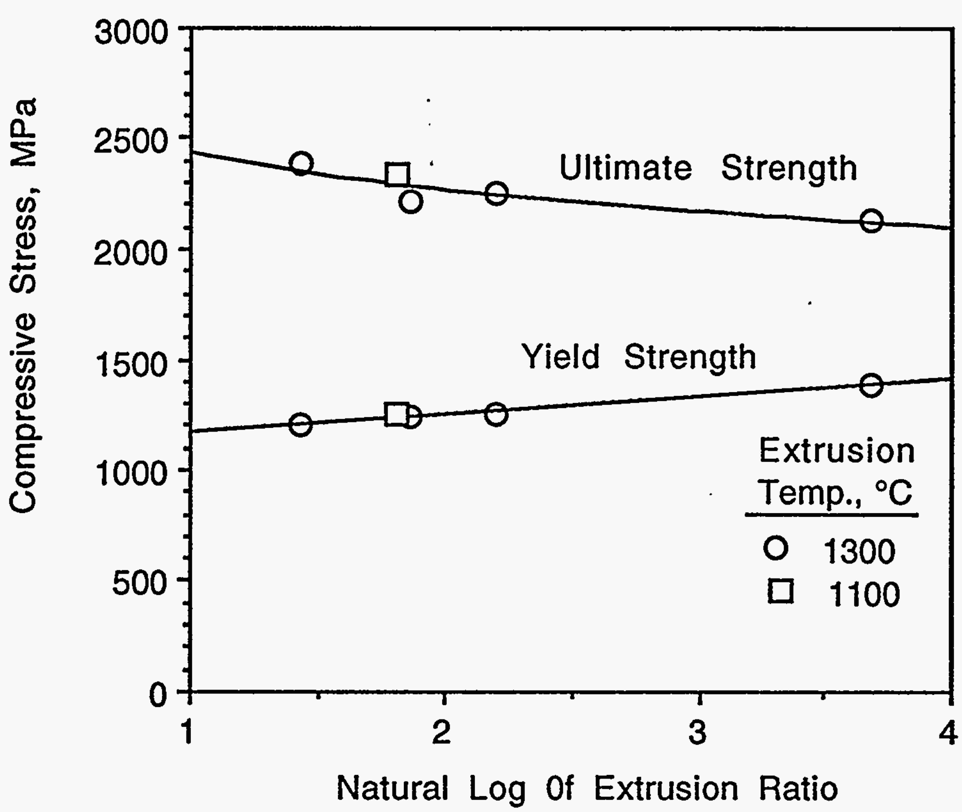

Figure 9. Compressive ultimate strength decreases and yield strength increases only slightly with increased extrusion ratio for tungsten-hafnium composites extruded at 1300°C preheat temperature.

2. A W-steel composite can be consolidated by hot extrusion to achieve full density at a 1000°C preheat temperature.

3. Metallographic examination of these materials shows elongated polycrystalline regions of tungsten within a continuous steel or hafnium phase and a highly distorted boundary between the two phases when viewed transverse to the rolling direction.

4. At a preheat temperature of 1300°C, there is complete recrystallization of the tungsten during extrusion of the W-Hf composite but only partial recrystallization at lower preheat temperatures. This is consistent with the temperature dependence of both the resistance to materials deformation, as measured by the extrusion constant, and the variation of RT microhardness and compressive yield stress with extrusion ratio.

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