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High temperature tensile properties and their application to toughness enhancement in ultra-fine grained W-(0-1.5)wt% TiC

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ABSTRACT

Ultra-fine grained (UFG) W-TiC consolidates are very promising for use as divertors in fusion reactors, however, the assurance of room-temperature ductility of UFG W-TiC remains unsettled. The assurance requires a sufficient degree of plastic working for the consolidates and thus overcoming of poor plastic workability in UFG W-TiC by applying superplasticity. Therefore, the magnitudes of elongation to fracture and flow stress which are important measures for plastic working were examined for UFG W-(0–1.5)%TiC (in wt%) at 1673–1973 K where superplasticity occurs without appreciable grain growth. It is shown that the elongation and flow stress are strongly dependent on TiC addition and atmosphere (Ar, H_2) during mechanical alloying (MA). As the TiC addition increases, the elongation significantly increases without appreciable increase in the flow stress level. W-TiC fabricated with MA in H_2 exhibits larger elongation and larger strain rate sensitivity of flow stress than W-TiC with MA in Ar. These results were applied to perform plastic working and the room-temperature bend test results for plastic worked W-1.0%TiC are shown.

1. Introduction

Tungsten (W) is very promising for use as divertors and structural materials exposed to irradiation environments; however, it exhibits serious embrittlement in several regimes, i.e., low temperature embrittlement, recrystallization embrittlement and radiation embrittlement [1]. It is thus required to develop W materials with improved resistance to such embrittlement.

Ultra-fine grained (UFG) W-TiC consolidates with equiaxed grains of 50–200 nm and nearly full densification of ~99% in relative density were recently developed by utilizing mechanical alloying (MA) [2] and hot isostatic pressing (HIP) [3–5]. They exhibited good resistance to irradiations with fast neutrons and helium-ions [4–6]. However, the assurance of room-temperature ductility of UFG W-TiC remains unsettled.

An important finding for the assurance is that the beneficial effects of plastic working after consolidation on fracture strength and room-temperature ductility for W-0.3%TiC (grain size:0.6–2 μm) become conspicuous with decreasing grain size [3,4]. In view of the much smaller grain size in UFG W-TiC, the beneficial effects of plastic working embrace the possibility of significant enhancement of room temperature ductility of UFG W-TiC.

W-TiC consolidates usually requires plastic working at much higher temperatures than the HIPing temperature, 1623 K, due to their poor plastic workability. At such high temperatures plastic working may cause significant grain growth, and the UFG structures cannot be maintained. Recently the authors found that UFG W-0.5%TiC consolidates exhibit superplastic deformation at temperatures where the UFG structures may be essentially maintained [5,7,8]. Superplastic deformation is expected to be applied to plastic working for UFG W-TiC consolidates. Important measures for plastic working are the magnitudes of elongation to fracture and flow stress, and thus it is necessary to clarify what are the factors that significantly affect superplastic behavior in UFG W-TiC and how the superplastic behavior varies by the factors.

In this study the effects of TiC additions and mechanical alloying (MA) atmosphere (Ar, H_2) on superplastic behavior were studied for UFG W-(0-1.5)%TiC consolidates. The results obtained were applied to plastic working and preliminary results for the plastically worked specimens of UFG W-1.0%TiC will be presented.

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2. Experimental

Powders of pure W (an average particle size 4.0 μm and purity 99.9%) and TiC (40 μm, 99.9%) were mixed to provide nominal compositions of W-0, 0.25, 0.5, 0.8, 1.1, 1.5TiC (in wt%) in a glove box. Each of the powder mixtures was then charged into two vessels made of TZM (Mo-0.5Ti-0.1Zr) together with TZM balls for MA. The inner atmosphere of the vessels for MA was a purified Ar or H_2 gas (purity 99.99999%). MA treatments were conducted by a 3MPDA (three mutually perpendicular direction agitation) ball mill. The details of the MA processes were reported elsewhere [3,9].

The MA processed powder was heated at 1073 K for 3.6 ks in a vacuum to remove the introduced Ar or H_2 gas during MA. The degassed powder was enclosed into a mild steel capsule and then subjected to HIP in an Ar atmosphere at 1620 K and 200 MPa for 10.8 ks. The dimensions of the as-HIPed consolidates were approximately 25 mm in diameter and 26 mm in height. As reported in previous papers, the use of an Ar atmosphere leads to slightly less densification and less impurity contents of Mo introduced from the TZM vessels and balls during MA [4,5,8]. Hereafter, W-(0-1.5)TiC consolidates fabricated with a purified H_2 or Ar atmosphere for MA are called W-(0-1.5)TiC-H_2 or W-(0-1.5)TiC-Ar.

Miniaturized tensile specimens with the gage sections of 1.2 mm by 0.5 mm by 5 mm [10] were machined and mechanically polished with emery disks up to #1500. Tensile tests were performed at temperatures from 1673 to 1973 K at initial strain rates of 5×10^{-5}–$5 \times 10^{-3}\ s^{-1}$ in a vacuum better than 5×10^{-4} Pa with Ta foils used for additional protection of the specimens from pick-up of gaseous impurities during the test. Details of the tests were reported elsewhere [11]. Fracture surfaces of the tested specimens were examined with a field emission scanning electron microscope (FE-SEM).

3. Results and discussion

Fig. 1 shows the tensile elongation at 1773, 1873 and 1973 K at a medium strain rate of $5 \times 10^{-4}\ s^{-1}$ for the HIPed compacts of W-(0-1.5)TiC-H_2 and W-(0-1.5)TiC-Ar as a function of TiC content. For the compacts without TiC addition very limited elongation (less than 10%) is recognized even at 1973 K regardless of MA atmosphere. However, it should be noted that the elongation significantly increases with increasing TiC addition. For W-TiC-H_2, only 0.25% TiC addition results in large elongation exceeding 160% at 1973 K, whereas for W-TiC-Ar such large elongation requires 0.8%TiC addition. The largest elongation is obtained at 1.1%TiC addition although the difference in elongation between 1.1 and 1.5%TiC additions is quite small. This suggests that approximately 1%TiC addition may be optimum for the purpose of toughness improvement by hot plastic working.

The present finding that the elongation of UFG W-TiC at 1673–1973 K is significantly improved by TiC additions is very important for performing the aimed amount of plastic working for poorly workable UFG W-TiC. Therefore, we discuss here the reason why TiC additions significantly increase the elongation and the elongation of W-TiC-Ar is less than that of W-TiC-H_2 even by 1.5%TiC addition.

Our previous study on W-0.5TiC-Ar showed that nano-sized Ar bubbles exist in the as-HIPed consolidates and the Ar bubbles coalesce and interconnect with each other by the applied stress, leading to fracture in a relatively small elongation [5,7,8]. Fig. 2 shows comparison of the specimen surfaces of W-0.5TiC-Ar and W-1.1TiC-Ar deformed at 1973 K and at $5 \times 10^{-4}\ s^{-1}$: W-0.5TiC-Ar fractures at an elongation of 55%, whereas W-1.1TiC-Ar does not fracture even after exceeding 160%. Cracks are distinctly observed in W-0.5TiC-Ar, but not in W-1.1TiC-Ar. This indicates that TiC additions have the beneficial effect of suppressing the formation of cracks and their interconnection.

Since TiC additions lead to grain size refining, grain boundary sliding is enhanced with increasing TiC addition. Fracture surface observations on the both specimens of W-0.5TiC-Ar and W-1.1TiC-Ar tested at 1973 K showed that although slight grain growth occurs during deformation, the equiaxed grain structures are still maintained. This implies that grain boundary sliding is the dominant deformation mechanism even for W-0.5TiC-Ar. On the other hand, grain boundary sliding results in stress concentration at triple points, leading to cracking preferentially at weak grain boundaries. Resistance to cracking can be improved by strengthening of the weak grain boundaries. TiC additions may strengthen the weak grain boundaries by forming TiC dispersoids at the boundaries and thus improve the resistance to grain boundary cracking.

The grain boundaries in W-TiC-Ar, in contrast to those in W-TiC-H_2, are considered to contain argon, which further weakens the grain boundaries and enhances the formation of cracks and their interconnection. As a result, in order for W-TiC-Ar to exhibit the elongation similar to W-TiC-H_2 more TiC additions were required than those for W-TiC-H_2.

Another important property for plastic working is the flow resistance. Fig. 3 shows the flow stress at 1773–1973 K for W-TiC-H_2 and W-TiC-Ar as a function of TiC addition under the same test conditions as in Fig. 1. It should be noted that the variation of

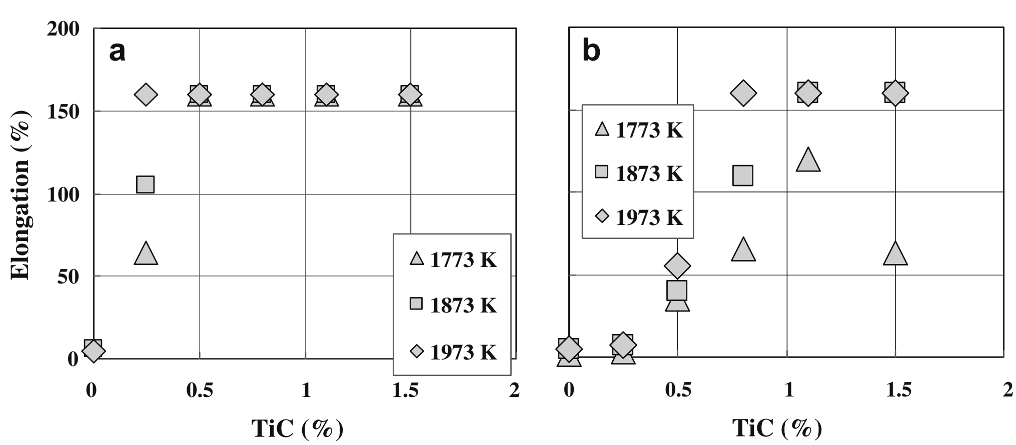

Fig. 1. Tensile elongation at 1773, 1873 and 1973 K at a medium strain rate of $5 \times 10^{-4}\ s^{-1}$ for the HIPed compacts as a function of TiC content: (a) W-(0-1.5)TiC-H_2 and (b) W-(0-1.5)TiC-Ar.

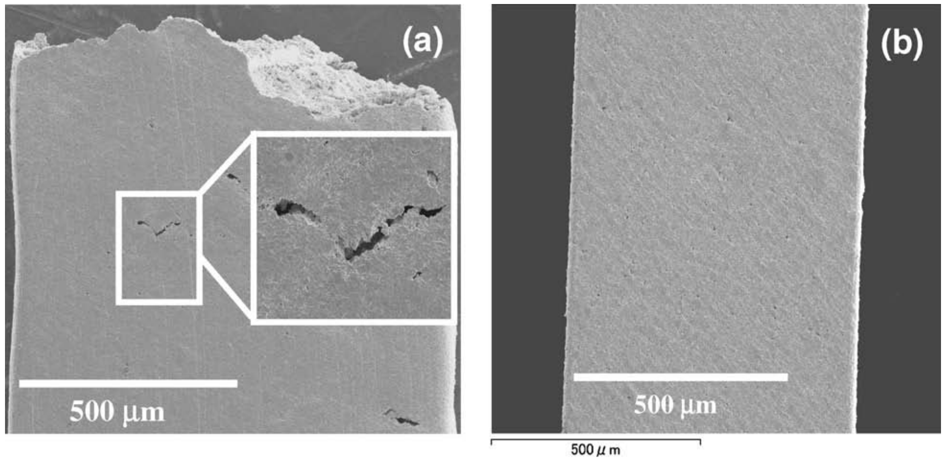

Fig. 2. Comparison of specimen surfaces of the HIPed compacts of (a) W-0.5TiC-Ar and (b) W-1.1TiC-Ar deformed at 1973 K and at 5×10^{-4} s^{-1}: W-0.5TiC-Ar fractures at an elongation of 55%, whereas W-1.1TiC-Ar does not fracture even after exceeding 160%. The tensile axis is vertical.

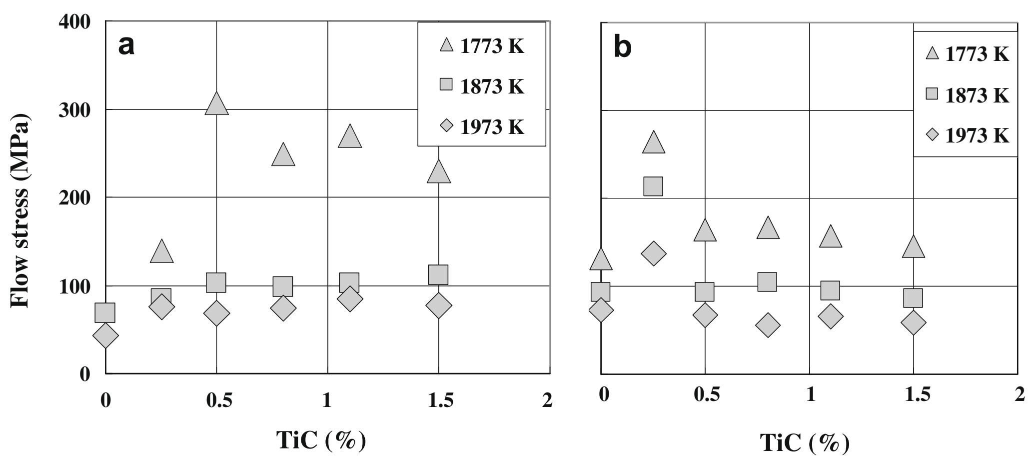

Fig. 3. Flow stress at 1773, 1873 and 1973 K at a medium strain rate of 5×10^{-4} s^{-1} for the HIPed compacts as a function of TiC content: (a) W-(0-1.5)TiC-H_2 and (b) W-(0-1.5)TiC-Ar.

the flow stress with TiC addition is small at all test temperatures, although an appreciable increase at 0.25%TiC for W-TiC-Ar is noticeable. This result suggests that the TiC phase up to 1.5% does not prevent grain boundary sliding.

Since plastic working is generally performed at higher strain rates than the present strain rates, the strain rate dependence of flow stress was examined to anticipate the flow stress level at the higher strain rates. It was found that good linear relationships are recognized between double logarithmic plots of flow stress and plastic strain rate under all the test conditions for W-(0-1.5)TiC-H_2 and W-(0-1.5)TiC-Ar. The slope of the linear relationship gives the strain rate sensitivity, *m*.

Fig. 4 shows effects of TiC addition on the *m*-value for W-TiC-H_2 and W-TiC-Ar. In this figure, the *m*-value is the average for the four temperatures since *m* showed very small temperature dependence. The *m* values are in a range between 0.4 and 0.5 for W-TiC-H_2 and between 0.2 and 0.3 for W-TiC-Ar, although the *m* values for W-1.5TiC-H_2 and W-0.25TiC-Ar are slightly smaller than 0.4 and 0.2, respectively. The variations of *m* with TiC addition are not significant. Comparison of the *m* values for W-(1.1, 1.5)TiC-Ar and W-(025, 0.5)TiC-H_2 which exhibit similar large elongation above 160%, shows that W-(1.1, 1.5)TiC-Ar exhibits clearly smaller *m* values than W-(025, 0.5)TiC-H_2 It is noted that such small *m* values are favorable for plastic working because they reduce the flow resistance for plastic working at the higher strain rates.

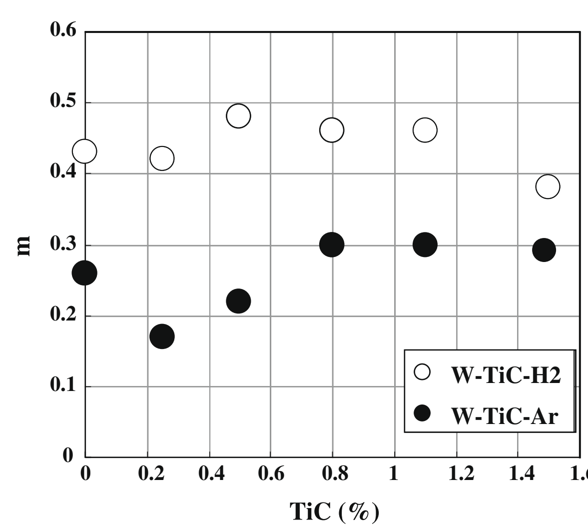

Fig. 4. Strain rate sensitivity of flow stress, *m*, for the HIPed compacts of W-(0-1.5)TiC-H_2 and W-(0-1.5)TiC-Ar as a function of TiC content.

The above results suggest that W-1TiC-Ar may be most suitable for plastic working because it exhibits the largest elongation

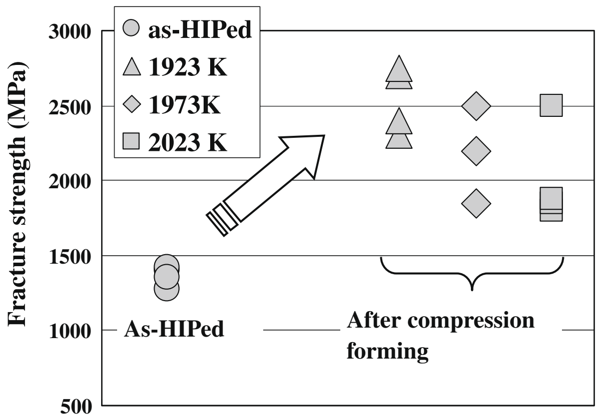

Fig. 5. Fracture strength of W-1.0TiC-Ar plastically worked by compression forming at 1923, 1973 and 2023 K together with that of as-HIPed W-1.0TiC-Ar.

among W-TiC-Ar as well as the smallest flow resistance in all the UFG W-TiC consolidates examined in this study. Therefore, the as-HIPed specimen of W-1.0TiC-Ar was subjected to plastic working. Relatively large degree of plastic working has been obtained by compression forming at 1923–2023 K, and three-point bending tests were performed at room temperature for W-1.0TiC-Ar with approximately 72–76% in compression ratio. The results are shown in Fig. 5. It is recognized that the fracture strength of W-1.0TiC-Ar is significantly increased by compression forming and the beneficial effect of compression forming tends to increase as the forming temperature decreases. Although the present result is preliminary one, it should be noted that W-1.0TiC-Ar with compression forming at 1923 K to 72% exhibits a very high strength of 2.75 GPa, compared with the as-HIPed ones, 1.3–1.5 GPa. In view of the aspect that effects of plastic working on toughness become prominent with decreasing the temperature of plastic working and probably increasing the rate of plastic working, further studies on compression forming under wide conditions should be needed and are in progress.

4. Conclusions

In order to clarify the effects of TiC additions and mechanical alloying (MA) atmospheres on superplastic behavior of UFG W-TiC, tensile tests were conducted at 1673–1973 K at strain rates of 5×10^{-5} to $5 \times 10^{-3}\ s^{-1}$ for UFG W-(0-1.5)%TiC consolidates with MA in a purified H_2 or Ar atmosphere. The results obtained were applied to overcome poor plastic workability in UFG W-TiC and preliminary results for the plastically worked UFG W-1.0%TiC were obtained. The main results and conclusions are as follows.

1. The high temperature mechanical properties (elongation to fracture, flow stress, *m*-value) that are essential to plastic working can be optimized by TiC addition and MA atmosphere (Ar, H_2).
2. The elongation significantly increases with increasing TiC addition. The flow stress and *m*-value exhibit less dependence on TiC addition.
3. Residual Ar results in decrease of elongation, flow stress and *m*-value. TiC addition offsets the reduction of elongation by Ar.
4. The major contribution of TiC is to suppress crack formation and its linkage at weak grain boundaries caused by grain boundary sliding.
5. 1%TiC addition is most likely suitable for plastic working. W-1%TiC-Ar specimens with compression forming at 1923 K to 72% exhibits a very high strength of 2.75 GPa, compared with the as-HIPed ones, 1.3–1.5 GPa.

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