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Discontinuously reinforced titanium matrix composites for fusion applications

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Abstract

We have reinforced α-Ti with different contents of TiC particles using the in situ technique and conventional casting. Compositional and microstructural characterization of the TiC/Ti composite material was made by XRD and SEM-EDS. Tensile tests at RT, 723 and 973 K have been performed on samples heat treated at 1000 K for 30 min which were prepared from cold rolled material. The effect of the content, size and morphology of the TiC particles on the tensile properties has been investigated. The results indicate that the expected improvement in the mechanical characteristics of TiC/Ti composites is inhibited by the detrimental presence of coarse dendritic particles of TiC. The premature failure of these composites at RT is due to cracking of the coarse TiC particles. Local softening due to inhomogeneous plastic deformation of the Ti matrix appears to contribute to the tensile failure of the TiC/Ti composites deformed at 723 and 973 K.

1. Introduction

The development of structural materials with good mechanical performance at elevated temperatures, a high resistance to radiation damage and a low induced long term activation is decisive to build reliable fusion reactors. Ti and its alloys are among the candidate materials due to their mechanical properties, corrosion resistance and fast decay rate after irradiation. For fusion applications, it is crucial to extend the operating temperature range of Ti base materials. The incorporation of hard particles into a Ti alloy matrix can be an efficient method to improve its mechanical properties at elevated temperatures [1–7]. Ti alloy matrix composites appear to have more promising mechanical behavior for conventional applications than Ti matrix composites [1,2,6,8–10]. However, in the environment of a fusion reactor Ti matrix composites could have a better behavior than the Ti alloy ones due to the detrimental effect in nuclear properties of alloying elements such as Al, Sn and B, and the instability of second phases under irradiation. In this sense it has been demonstrated that α-alloys exhibit better stability under irradiation as compared with the $\alpha + \beta$ alloys [11].

So far, the use of discontinuously reinforced composites is limited by their fabrication and processing difficulties. In situ reinforced Ti matrix composites produced by casting, compared to those produced by other techniques, offer a low cost and possibilities to improve remarkably their mechanical properties and temperature capabilities by thermomechanical treatments. Particles of a hard phase, such as TiC, in a Ti matrix cannot be sheared by gliding dislocations and remain stable in size and distribution up to the highest service temperature of the material. Graphite in a Ti melt reacts exothermally to form a dispersion of submicroscopic TiC particles. After solidification, these

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particles, homogeneously distributed in the matrix, tend to be very stable through subsequent processing and the use of the material at elevated temperatures would be feasible. Since strain accommodation across the TiC/Ti interface is possible by dislocation generation, according to transmission electron microscopy observations [5], it is expected that the as-cast composite will be significantly strengthened. In addition, strength enhancement could be developed by subsequent thermomechanical treatments.

Ti matrix composites in situ reinforced with TiC particles have been investigated in an attempt to assess their capability for fusion applications. At present, there are few reports in the literature on the mechanical characteristics of TiC/Ti composites prepared by casting techniques.

2. Experimental procedure

The required amounts of 99.5% pure sponge Ti and 99.9% pure graphite powder were blended and milled for 5 h at 525 rpm in a planetary ball mill. The vials were charged and sealed in a high-purity Ar atmosphere. The milled material was compacted into pellets of about 12 g. TiC/Ti composite buttons with a TiC content of less than 10% in weight were prepared by repeated arc melting of these pellets in a high-purity He atmosphere. Flat slices perpendicular to the bottom of the buttons were cut and cold rolled up to a thickness reduction of about 20% in order to prepare samples with suitable dimensions for tensile tests. Rolled slices showing no evidence for cracks were electro-discharge machined to prepare tensile test samples with gauge dimensions of $8.0 \times 2.5 \times 0.4$ mm^3. These samples were heat treated at 1000 K for 30 min followed by air cooling to room temperature in vacuum ($\leqslant 10^{-3}$ Pa). The composite samples were chemically analyzed by X-ray diffraction (XRD), and the TiC fraction was determined from the C content measured by absorption in the IR range using a LECO equipment. Typical densities of Ti and TiC were used to calculate the volume fraction of composites. The microstructure of the samples were examined by scanning electron microscopy (SEM-EDS) and optical microscopy. Tensile tests at room temperature, 723 and 973 K were carried out at a strain rate of 4.2×10^{-5} s^{-1} in a Zwick machine equipped with a furnace. The high-temperature tests were done under flowing high-purity Ar. Pure Ti samples, produced from the same sponge material, and samples of a commercial TiC 10 vol.%/Ti–6Al–4V composite (CermeTi-10C) supplied by Dynamet Technology Inc., were tested for comparison. The Ti samples underwent the same processing and heat treatment than the TiC/Ti composite samples but the Ti–6Al–4V matrix composite ones were tested in as-received state.

3. Results and discussion

3.1. Microstructure and tensile behavior

XRD measurements in the composites indicate the presence of a single Ti carbide phase with NaCl (B1) structure, identified as TiC_x. From the measured lattice parameters the composition is estimated to range between $TiC_{0.5}$ and $TiC_{0.6}$. The results indicate a trend to a decrease in the stoichiometry of the TiC particles as the TiC fraction increases in the composite. Figs. 1 and 2 show the SEM images of as-prepared and tensile tested composites. In the as-cast samples the TiC reinforcements are found uniformly distributed showing two different morphologies. The coarser particles (mean diameters ~6–10 μm) have a dendritic aspect while the finer ones (mean diameters < 6 μm) are equiaxed or near equiaxed. It should be noted that coarse and dendritic particles were not observed in the 2.1 vol.% composite (see Fig. 1), but they appear and are more abundant in the material as the fraction of TiC increases. This result along with the XRD measurements suggests that the fine and equiaxed, or near equiaxed, particles have a composition close to $TiC_{0.6}$ while the composition for the

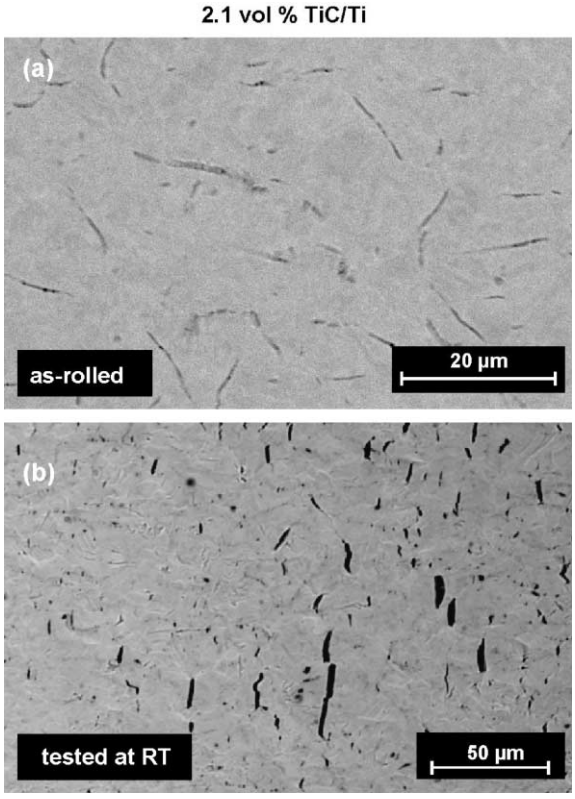

Fig. 1. SEM images of 2.1 vol.% TiC/Ti: (a) cold rolled and (b) tensile tested at room temperature.

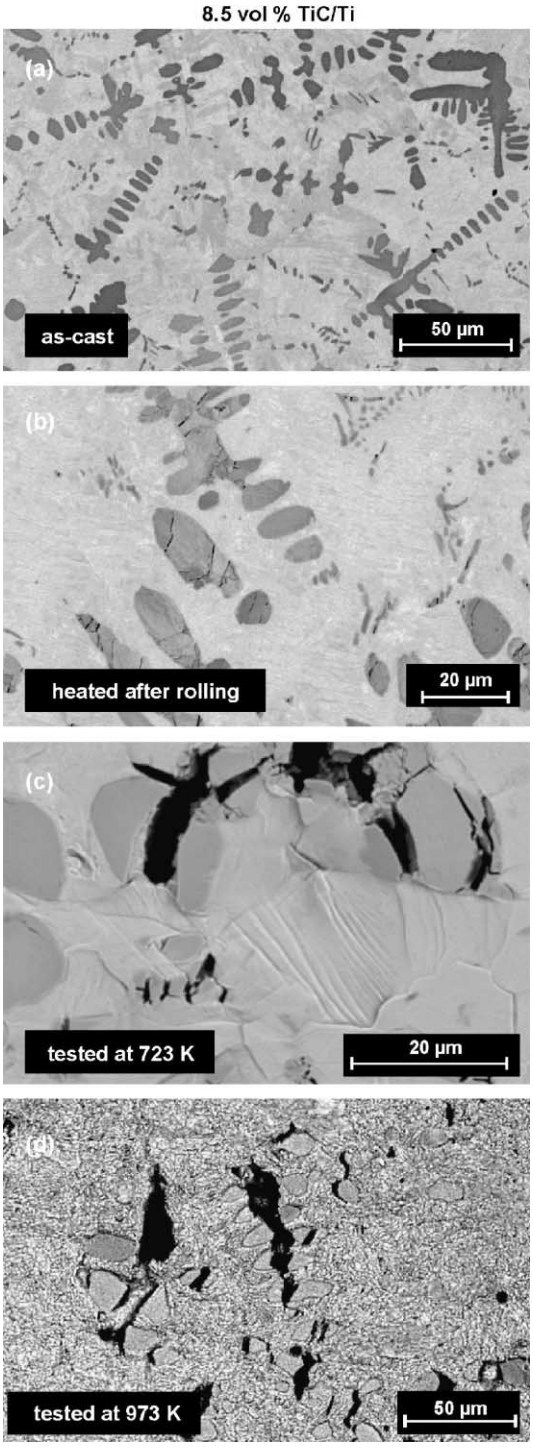

Fig. 2. SEM images of 8.5 vol.% TiC/Ti: (a) as-cast and (b) thermal treated at 1000 K after cold rolling. Crack-like cavities in TiC particles after tensile testing at (c) 723 K and (d) 973 K.

coarse and dendritic carbide should be $TiC_{0.5}$. Since the Ti–C phase diagram is eutectic, in addition to primary dendritic TiC particles formed in undercooled liquid Ti, a second precipitation of TiC must occur by eutectic decomposition during cooling down from the eutectic temperature. These TiC particles precipitated by the eutectic reaction would correspond to the finer and equiaxed ones present in the TiC/Ti composites [12]. Cold rolling and the subsequent heat treatment appears to be effective in breaking up the particles, as reveals Fig. 2(b), and producing finer sizes.

Table 1 shows the tensile properties of the TiC reinforced composites along with the ones for unreinforced Ti and CermeTi-10C. Representative stress–strain curves for tensile tests at room temperature and 723 K are shown in Fig. 3. The strengthening by TiC dispersion at RT (Fig. 3(a)) is evident as expected, although the strength of these composites does not exhibit a significant dependence with the TiC content. However, the 2.1 vol.% TiC composite appears to be remarkably more ductile than the composite material with a higher reinforcement content. This ductility improvement is attributed to the absence of dendritic particles and smaller size of the reinforcements. Fig. 3(b) shows the results at 723 K. As in the previous case, TiC composites show an increase in yield and ultimate stresses, as compared with pure Ti, with a decrease in the ultimate elongation except for the case of 2.1 vol.% TiC composite. Stress values decrease significatively at 973 K, only ultimate stresses are shown in the table, and all the TiC composites exhibit higher ductility than pure Ti.

3.2. Plastic instability

All TiC/Ti and CermeTi-10C samples deformed at 723 K exhibit a serrated stress–strain curve. At 973 K, a significative serrated flow on the stress–strain curve only is observed for 8.5 vol.% TiC/Ti and CermeTi-10C, see Fig. 4. Ti samples deformed under the same conditions do not show any evidence for serrated flow. Therefore, the TiC particles or the C present in the matrix appear to be responsible for plastic instabilities causing the observed serrated flow. The origin of these instabilities has not been specifically investigated but it produces inhomogeneous deformation of the samples. The striations observed in these samples reveal the formation of deformation bands localized in some grains giving rise to a local softening in the matrix and the premature failure of the sample, as shown in Fig. 2(c). It is known that flat samples under tensile test can develop inhomogeneous deformation.

3.3. Failure characteristics

Three different types of cavities can be observed near the fracture area. In all the tested samples of TiC/Ti, crack-like cavities through the particles are extensively observed in the coarse and middle size TiC particles, see

Table 1
Tensile properties for pure Ti, TiC/Ti composites and CermeTi-10C tested at a strain rate of 4.2×10^{-5} s^{-1}

TiC (vol.%)	298 K			723 K			973 K		
	YS (MPa)	UTS (MPa)	ε_u (%)	YS (MPa)	UTS (MPa)	ε_u (%)	YS (MPa)	UTS (MPa)	ε_u (%)
0.0	230	386	12.4	43	85	10		19	30
2.1	414	565	18.5	133	271	17		28	53
3.4	437	514	2.3	81	246	10		32	45
3.5			1.7	101	190	5.5		25	44
4.1	418	516	2.8					31	49
5.7	346	405	2.2	148	220	2.5		30	48
7.6				163	215	3.6		36	24
8.5	405	474	2.1	49	194	6.0		37	44
CermeTi-10C	617	617	0.9	87	523	4.1	50	184	6.9

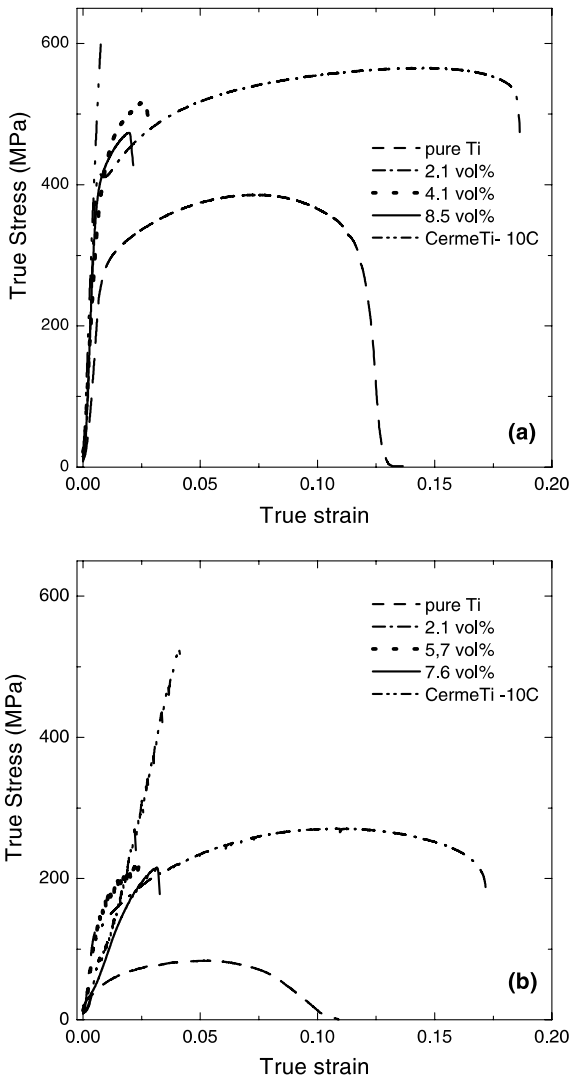

Fig. 3. Stress–strain curves for samples tested at (a) room temperature and (b) 723 K.

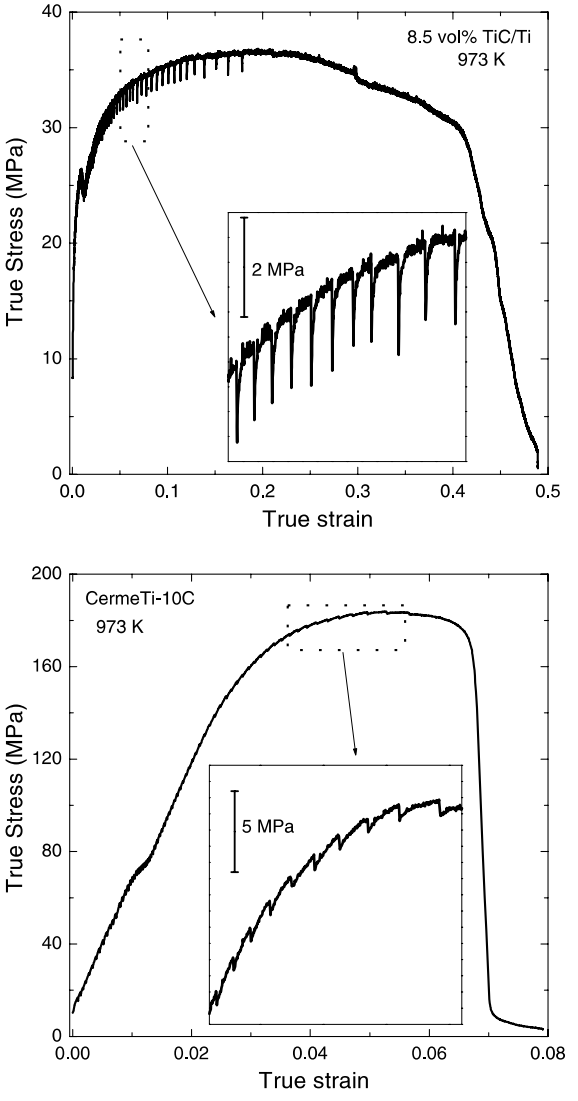

Fig. 4. Stress–strain curves showing serrated flow for 8.5 vol.% TiC/Ti and CermeTi-10C tested at 973 K.

Fig. 2. These cavities are nucleated on the microcracks produced in the particles during the rolling process, and they grow transversely to the tensile axis under deformation. A second type of cavities are also formed on the interfaces between the matrix and the reinforcement. This is evident for samples having coarse particles when they are deformed at 973 K as shown in Fig. 2(d). This points out the possible debonding between the TiC particles and the Ti matrix at elevated temperatures. Fig. 2(d) also shows that it can appear void formation in the Ti matrix at high temperatures.

In samples with a high density of reinforcing particles, or particle clusters, cracks are formed by coalescence of crack-like cavities (first type). The tensile failure at room temperature and 723 K appears to be produced in a transgranular and intergranular brittle way when cracks initiating at the cavities propagate to the Ti matrix. The clustering and size of the particles appears to control the crack growth and failure. This accounts for the premature failure of the composites which have a coarse or clustered dispersion of reinforcing particles, and the better behavior of the 2.1 vol.% composite. All the TiC/Ti samples exhibit a significative ductility at 973 K, higher than the one for the Ti sample, and a ductile dimpled failure. This ductility would be caused by the void formation in the Ti matrix.

All the tensile tested samples of CermeTi-10C show TiC particle cracking, and cracks in the alloy matrix developed from cracks initiated in the cracked TiC particles as shown in Fig. 5. It should be noted that the sizes of the TiC particles in CermeTi-10C are remarkably larger (~20 μm) than those observed in the TiC/Ti composites. It appears that cracking along the reinforcement colonies controls the failure of this material. In contrast to the results for TiC/Ti composites, tensile deformation in CermeTi-10C samples does not promote cavitation inside the cracks of the TiC particles.

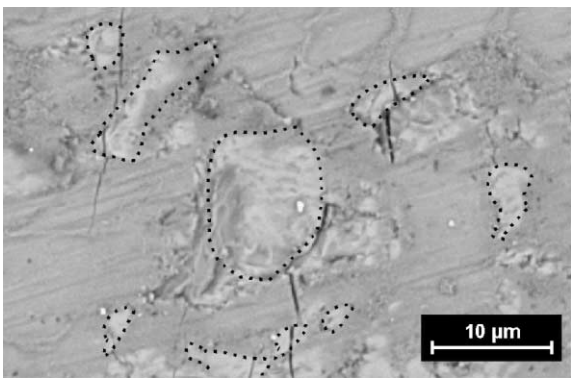

Fig. 5. CermeTi-10C tensile tested at 973 K showing the initiation of cracks in the matrix. Dotted curves mark the contour of TiC particles.

4. Conclusions

α-Ti in situ reinforced with TiC using a conventional casting technique exhibits a dual morphology consisting of coarse dendritic particles and fine particles with equiaxed and near-equiaxed shapes. The tensile test results indicate that the presence of coarse TiC particles deteriorate the mechanical properties of the composite material. The optimum vol.% seems to be in the range 2.1–3.4%.

The premature tensile failure of these TiC/Ti composites at RT is due to cracks produced by coalescence of crack-like cavities generated in the TiC particles. Tensile deformation in the range 723–973 K can produce an inhomogeneous distribution of the plastic deformation causing the local softening and premature failure of the composite material. These plastic instabilities, which are revealed by serrated stress–strain curves, could be propitiated by the particular geometry of the samples used in the tensile tests.

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