研　　究

Microstructure and Properties of γ-TiAl/Ti_2AlC Composites Produced by Reactive Processing from Elemental Powders

Rajagopalan Ramaseshan☆[1,2], S. Krishnamoorthy Seshadri☆[2], N. Gopalakrishna Nair☆[2], Hiroshi Tsuda☆[1], Hiroshi Mabuchi☆[1] and Kenji Morii☆[1]

☆[1]Department of Metallurgy and Materials Science, College of Engineering, Osaka Prefecture University, 1-1 Gakuen-cho, Sakai 599-8531.
☆[2]Indian Institute of Technology, Madras - 600 036. India.

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SYNOPSIS

The γ-TiAl/Ti_2AlC composites with and without impurities, Ni, Cl and P, were prepared by combustion reaction from the elemental powders and homogenized after arc melting. The resulting composition is about 18 vol% Ti_2AlC in the matrix of TiAl with a lamellar structure of Ti_3Al (α_2). In the homogenized specimens, the α_2 phase transforms to the precipitated Ti_2AlC particles in the place of solutionizing lamellar structure. The composite material has a high strength both at ambient and elevated temperatures; about 800 and 400 MPa, respectively, with an ambient temperature ductility of 0.7% at bending test. The fracture toughness test also proves the composite homogenized is better than the as cast and value is 17.8 $MPam^{1/2}$. The crack propagation in the homogenized composite is zigzag mode and the precipitated particles are main obstacles for the crack propagation. The composite with impurities shows a marginal improvement in the oxidation resistance over the composites without impurities.

KEY WORDS

TiAl/Ti_2AlC composite, combustion reaction, dispersion strengthening, fracture toughness, oxidation resistance

1 Introduction

In recent years intermetallic phases have become a subject of intensive research with the aim of developing new materials which can be used in wide range of high temperature applications. Particular attention has been paid to the aluminides especially γ-TiAl which is one of the most promising candidate due to its low density, high melting point, excellent corrosion and oxidation resistance when compared to Ti_3Al (α_2) and orthorhombic (Ti_2AlNb) classes of aluminides. The major problems limiting the practical use of this compound are their poor ductility and formability at ambient temperatures with low oxidation resistance above 800°C. The ductility of certain intermetallics, such as titanium aluminides, has been improved considerably by a combination of alloying and thermo-mechanical processing[1]. In terms of oxidation and embrittlement, the γ-class of titanium aluminides looks promising for applications below 800°C [2]. Intermetallic composites could provide the right combinations of high-temperature strength, creep resistance, environmental stability with adequate ambient temperature ductility and low density. Such a composite with good oxidation resistance and high strength at elevated temperatures would extend its potential for engine component applications such as high pressure compressor, turbine and to some combustor applications.

Many composites have been studied with ductile particle reinforcements like β-TiNb[3,4] and with brittle particle reinforcements TiB_2[5,6], Ti_2AlC[7-11], Ti_2AlN[12], TiB_2+Ti_2AlN[13] and TiB_2+Ti_2AlC[14]. The ductile reinforcements improve the toughness of the composites and the brittle ones improve the strength, both at ambient and elevated temperatures. Particulate reinforcements and in-situ precipitation offer good result for exploitation in the short term applications[15]. The compatibility problems between titanium alloys[16], Ti_3Al[17] and TiAl[15,17] and its carbides are very less, especially TiAl/Ti_2AlC[7]. Because of the dispersed particulate reinforcements and/or the finely precipitated particles, these composites are much more resistant to recrystallization and grain growth than single phase alloys. However, for elevated temperature applications the oxidation resistance of this composite should be enhanced.

The β-stabilizers of titanium effectively promote the formation of the aluminum oxide[18] that is very much essential to protect the matrix material from oxidizing environment above 800°C than the mixed oxides of TiO_2+Al_2O_3[2]. The oxidation resistance of these intermetallic alloys[19,20] and its composites[21] were improved marginally by the addition of impurities like Cl and P. The addition of P to the elemental powders are possible only by the electroless process[22]. Hence, single phase γ-TiAl with the addition of P and Cl was taken up for investigation. Composites of γ-TiAl reinforced with Ti_2AlC and containing small amounts of above said impurities were studied for the fracture toughness and cyclic oxidation resistance.

2 Experimental procedure

High purity elemental powders of Ti (99.9%, –350 mesh; containing 3500 ppm level of oxygen), Al (99.9%, –150 mesh), C (graphite type, 99.99%, –400 mesh) were taken to prepare the intermetallic composites of γ-TiAl/Ti_2AlC. Initially the elemental titanium powders were given a coating of nickel by electroless process, discussed elsewhere[22]. The electroless nickel coating given to the titanium powder, deposits Ni, P and Cl, which are used to increase the oxidation resistance of the composite. Hereafter, the above said elements Ni, P and Cl are referred to as impurities. The Ni addition to the matrix was maintained well below 1 at% to avoid the Laves phase formation, which ultimately weakens the composites by reducing its ductility. The coated titanium powder and Al, C powders were mixed thoroughly for further processing with an initial composition of $Ti(Ni)_{50}Al_{45}C_5$ (in at%). The elemental powders composition for the γ-matrix, with and without impurities are shown in the Table 1. The vol% of the reinforcement was calculated as about 18% from the microstructure and the density[7]. In this process the mixtures of powders are compacted and subjected to exothermic synthesis for a duration of 10 min in a silica tube at a temperature 1000°C under a vacuum of ~10^{-4} Pa. The combustion reaction process has been successfully used to prepare the composite, Ti_2AlC reinforced γ-TiAl. The reactive processed materials of high porosity were arc melted repeatedly (two or three times), under Ti gettered high purity argon, in order to obtain high density composites. In Table 2 the measured composition of the ingots after processing is shown.

Figure 1 shows the binary phase diagram[23] of TiAl at the two phase region of α_2+γ. The as cast composites were annealed at 1000°C for 144 h to homogenize them. The as cast and homogenized composites were cut at a dimension of 3×3×5, 20×3×2, 20×3×3, and 10×3×2 mm^3 for compression, bending, fracture toughness and oxidation resistance tests, respectively. For fracture toughness test, the samples were prepared with a U-notch on the sample, polished and etched. The U-notch width was maintained at 0.15 mm and length at approximately 0.3~0.4 mm. The as cast and homogenized composites were characterized using optical and transmission electron microscopy.

Table 1 Nominal compositions of the alloys used, and the estimated matrix composition and the calculated volume fraction of reinforcement, Ti_2AlC.

Powder (at%)	Matrix (at%)	Reinforcement (vol%)
$Ti_{50}Al_{45}C_5$	$Ti_{50}Al_{50}$	~ 18
$Ti(Ni)_{50}Al_{45}C_5$	$Ti_{50}Al_{50}$	~ 18

Table 2 Measured compositions (in at%) for the matrix with and without impurities.

Alloy (homogenized)	Ti	Al	Ni	Cl	P
$Ti_{50}Al_{45}C_5$	50.89	49.11	–	–	–
$Ti(Ni)_{50}Al_{45}C_5$	50.45	49.27	0.13	0.03	0.12

3 Results and discussion

3.1 Microstructures

The composite formed by the combustion synthesis had a matrix of two phase α_2+γ, the reinforcement particles Ti_2AlC and had some porosity. Subsequent to arc melting the composite retained the matrix and reinforcement as before with significant reduction in porosity to obtain a high density composite. Figures 2(a)~2(d) show the microstructures of the composites of as cast and homogenized at 1000°C with and without impurities. Figures 2(a) and 2(c) are as cast structures and the rest

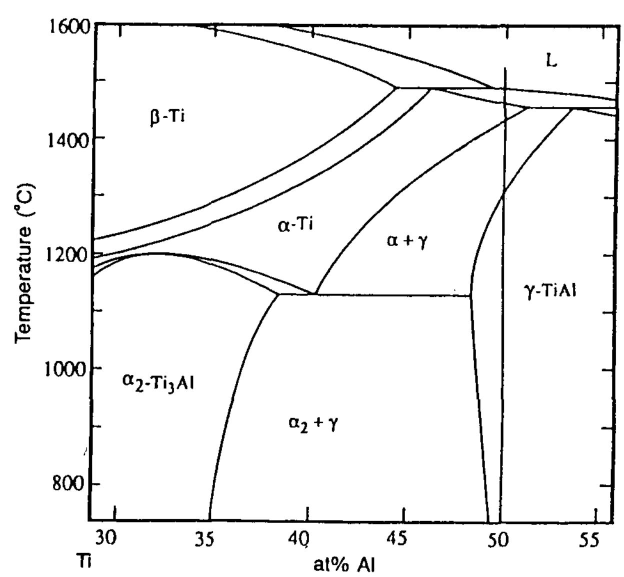

Fig.1 The central portion of binary Ti-Al equilibrium phase diagram[23] showing estimated matrix composition and homogenizing temperature for the alloys used.

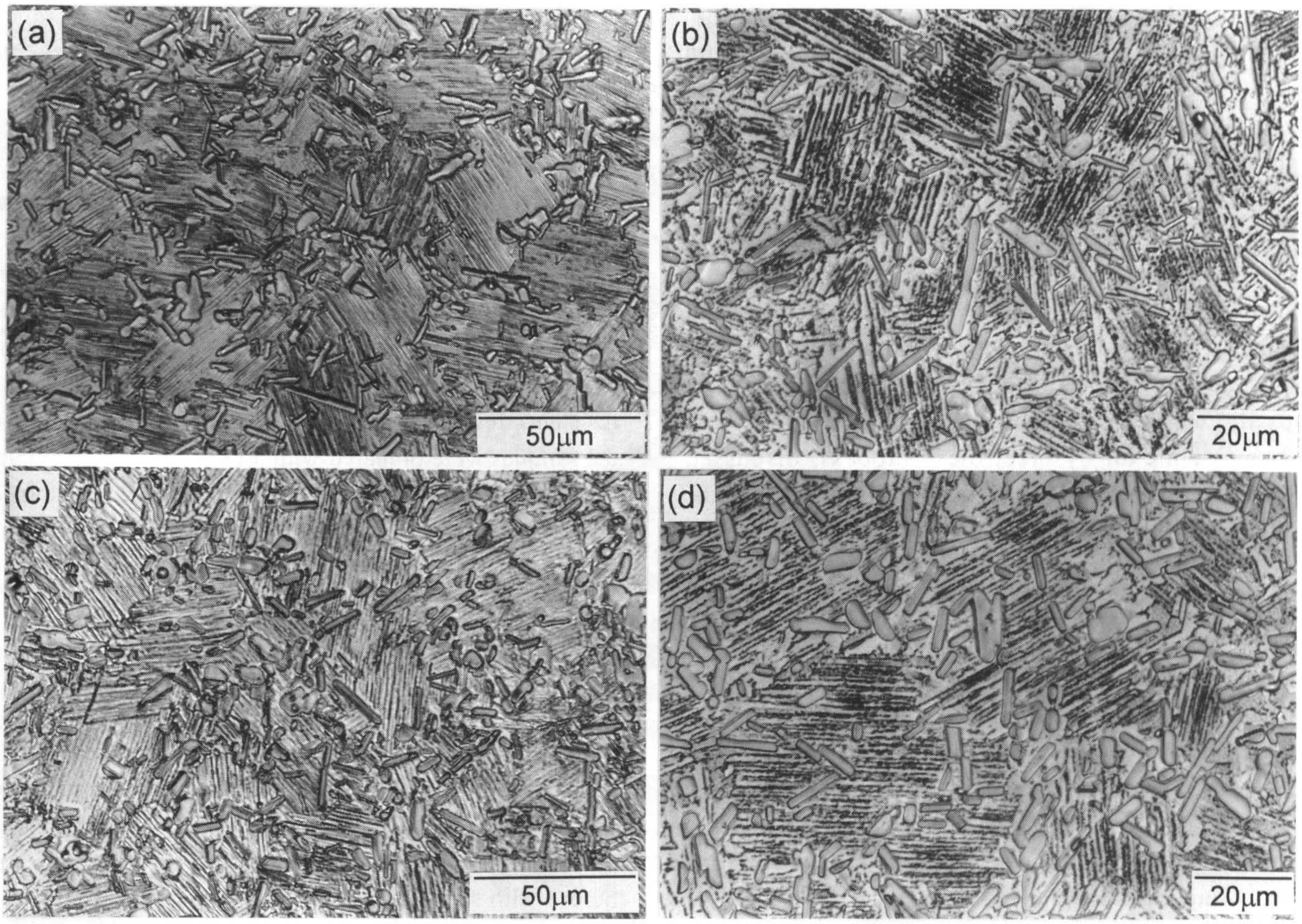

Fig.2 Optical microstructures of the arc-melted composites; (a) as cast and (b) homogenized composites without impurities ($Ti_{50}Al_{45}C_5$), and (c) as cast and (d) homogenized composites with impurities ($Ti(Ni)_{50}Al_{45}C_5$).

(Figs.2(b) and 2(d)) are homogenized at 1000°C for 144 h. The α_2 phase, nearly lamellar structure, is formed during cooling from liquidus encountered during above process. The α_2 phase titanium aluminide can dissolve carbon up to about 2 at% whereas γ–phase can dissolve less than about 0.5 at%, from the Ti-Al-C phase diagram[24] that is shown in Fig.3. Mabuchi et al. has shown that the carbon soluted α_2 matrix is very brittle compared to pure α_2 phase[11].

During homogenizing process α_2 phase containing carbon is transformed to Ti_2AlC particles by precipitation (Fig.2(b) and 2(d)). The TEM micrographs taken at the lamellar positions before and after homogenizing also reveal the precipitates, are shown in Figs.4(a) and 4(b). The precipitated particles may be acting as a crack arrester during fracture (as can be seen in Fig.8). There is no change in the microstructure of the composites with and without impurities. As shown in the optical micrographs, particles of random size are uniformly distributed in the matrix. The aspect ratio of the reinforcement Ti_2AlC particle is 5–10 and the precipitate size is about 0.3 μm.

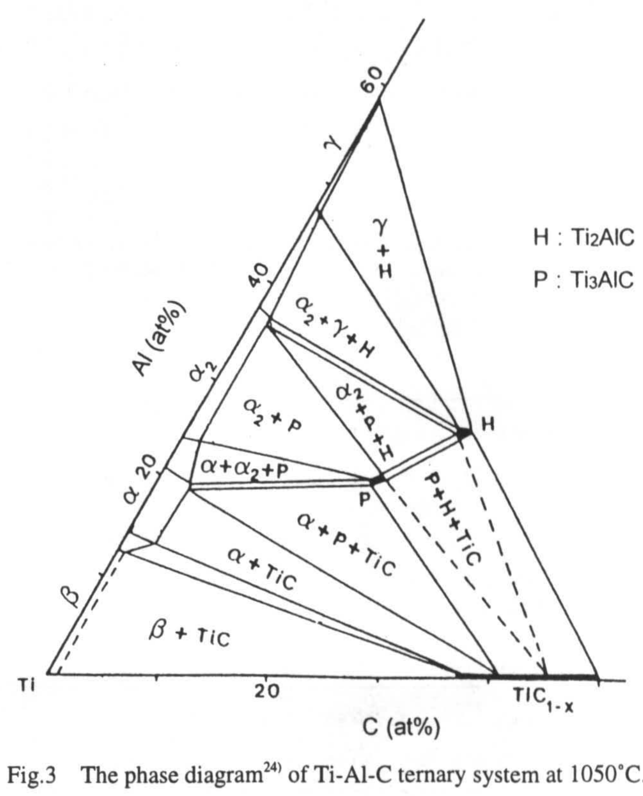

Fig.3 The phase diagram[24] of Ti-Al-C ternary system at 1050°C.

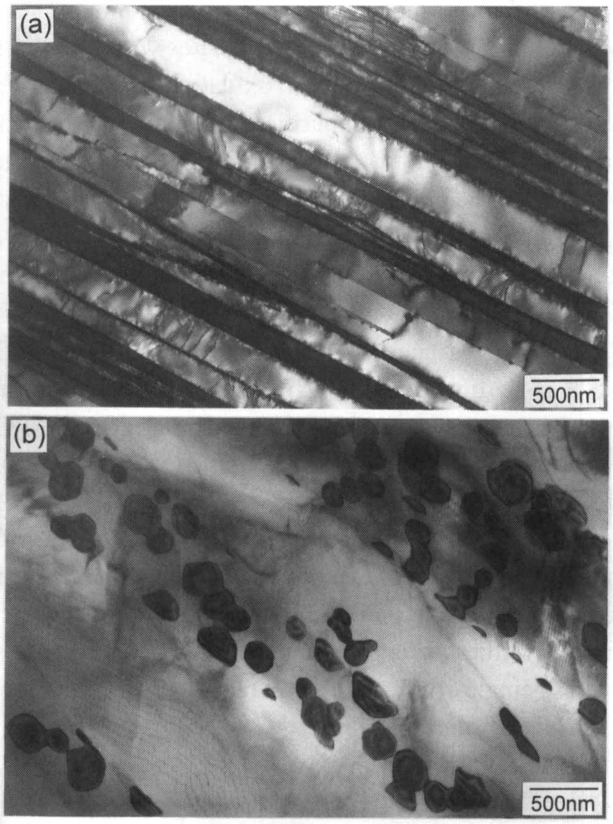

Fig.4 TEM micrographs of the composite with impurities ($(Ti(Ni))_{50}Al_{45}C_5$), showing smaller precipitates in place of lamellar disappeared; (a) as cast, and (b) homogenized at 1000°C.

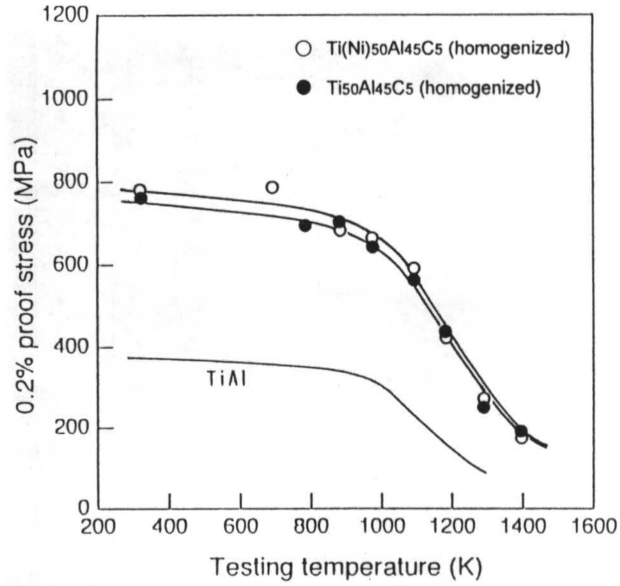

Fig.5 Compressive 0.2 % proof strength as a function of the testing temperature. Included for purpose of comparison is the proof strength of binary TiAl alloy.

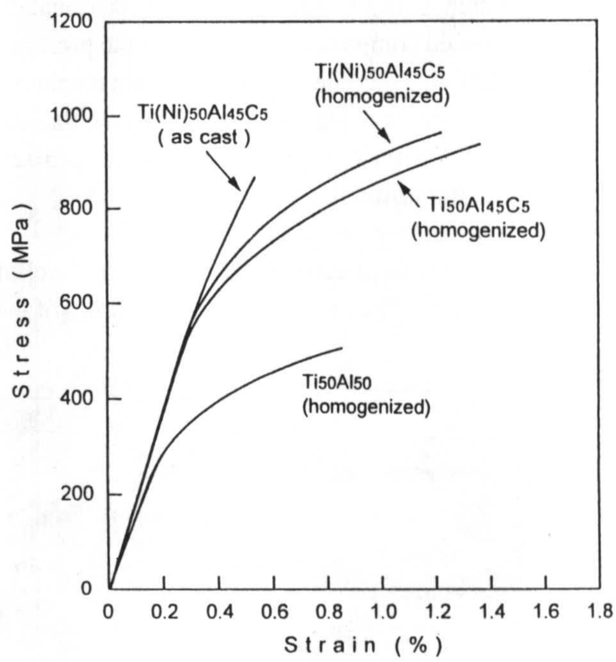

Fig.6 The stress-strain curves of bend tests at ambient temperature.

3.2 Strength and ductility

Figure 5 shows the 0.2% proof strength of the composites and un-reinforced matrix TiAl homogenized at 1000°C for 144 h during compression test. The composite with impurities shows a slight improvement over the composite without impurities. The strength of the composites decreases with the temperature as in the case of the un-reinforced γ-matrix. The proof stress values decreases gradually at low temperatures and rapidly at high temperatures indicating the dislocation activation at high temperatures. The nickel addition to the matrix improves the strength of the composite. But at higher temperatures there is no difference between the composites with and without impurities.

Figure 6 shows the ductility of the composites and the matrix TiAl during bending test. The ductility of the composite containing impurities (homogenized) is lower than the composite without impurities (homogenized). This is because of the impurities that are substituting the titanium or aluminum element in the lattice positions. The composites with impurities show 0.7% ductility which is more than that for γ-TiAl. The strength and ductility of as cast composite are low because of the carbon dissolved α_2 lamellar structure makes which the composite brittle. The modules of the composites (both homogenized and as cast) are look similar because of the reinforcement effect.

3.3 Fracture toughness

Fracture toughness tests have been carried out at ambient temperature on γ-TiAl/Ti_2AlC composites, as cast and annealed at 1000°C. Figures 7(a) and 7(b) show the crack propagation in the composites with impurities (as cast and

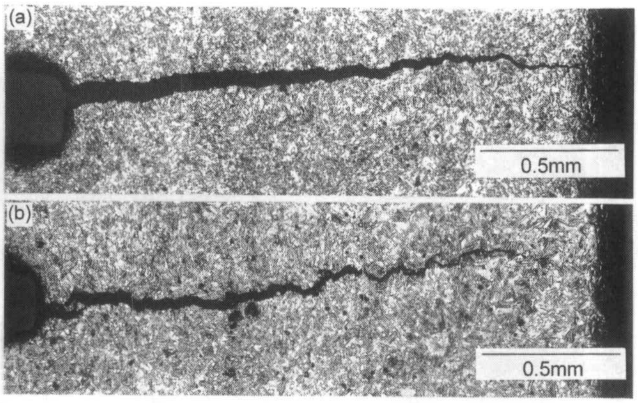

Fig.7 Optical micrographs of the fracture toughness tested composites with impurities ($Ti(Ni)_{50}Al_{45}C_5$); (a) as cast, and (b) homogenized at 1000°C.

homogenized specimens, respectively) during three point bend test. The crack propagation in the homogenized composites appears to be zigzag unlike the as cast composites which is linear. The zigzag crack propagation in the homogenized composites is because of the presence of precipitated Ti_2AlC particles (Fig.8). Fracture toughness values are presented in Table 3 and it can be seen that the best value, 17.8 $MPam^{1/2}$, is obtained for homogenized composite with impurities.

3.4 Oxidation resistance

High temperature applications demand a high level of oxidation resistance. The oxidation rate (mass gain) of the matrix and the composites at 900°C in air under cyclic oxidation is shown in Fig.9. Composites with impurities show marginal improvement in oxidation resistance over the composites without impurities. This is because of the presence of Cl and P that acts as promoters of Al_2O_3 in this composite. The presence of Cl forms $AlCl_3$ which assists in bringing Al to the surface of the composite. The P addition effectively reduces the oxygen vacancies in the TiO_2, resulting in the suppression of the inward diffusion of oxygen via vacancies[20]. The combined effect of the Cl and P is to improve the oxidation resistance of the composites at 900°C in air by enhancing the external Al_2O_3 formation in preference to mixed oxide layer of TiO_2 and Al_2O_3.

There is hardly any mass gain of the composites with impurities after 20 h of exposure at this temperature. This suggests the formation of a highly adherent layer which prevents further oxidation and spalling. However, further work is being carried out to study the effect of impurity on oxidation resistance.

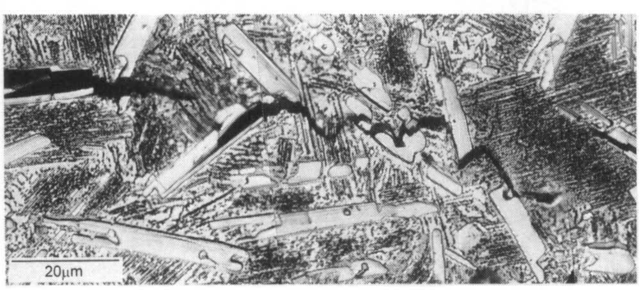

Fig.8 An optical micrograph taken at the crack tip of the composite with impurities ($Ti(Ni)_{50}Al_{45}C_5$) homogenized at 1000°C.

Table 3 Fracture toughness at ambient temperature of the composites with and without impurities.

Alloy	KIC ($MPam^{1/2}$)
$Ti_{50}Al_{45}C_5$ (as cast)	12.0
$Ti_{50}Al_{45}C_5$ (homogenized)	14.0
$Ti(Ni)_{50}Al_{45}C_5$ (as cast)	13.6
$Ti(Ni)_{50}Al_{45}C_5$ (homogenized)	17.8

4 Conclusion

During homogenizing the carbon dissolved in the matrix transforms to the precipitated particles. The precipitated carbide particles are the main obstacles to the crack propagation rather than the carbide particles formed during combustion reaction, thereby enhancing ductility and

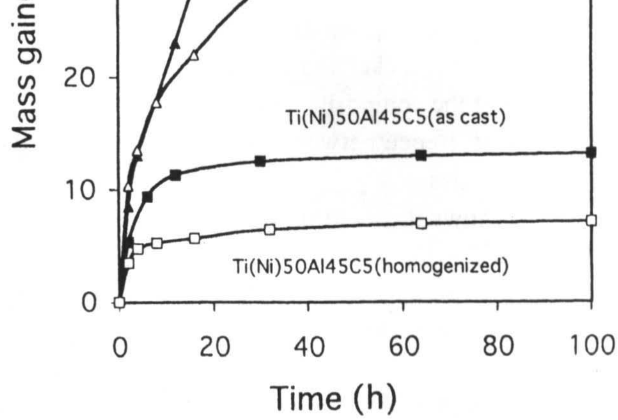

Fig.9 Mass gain curves as a function of oxidation time at 900°C in air.

fracture toughness. Composites containing impurities, both as cast and homogenized, show very good oxidation resistance.

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