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Improvement of the creep properties of TiAl alloys densified by Spark Plasma Sintering

Houria Jabbar [1], Jean-Philippe Monchoux [1], Marc Thomas [2], Florian Pyczak [3] and Alain Couret [1 *]

1 : CNRS ; CEMES (Centre d'Elaboration de Matériaux et d'Etudes Structurales) ; BP 94347, 29 rue J. Marvig, F-31055 Toulouse, France
2 : DMSM/ONERA, 29 Avenue de le Division Leclerc, BP 72, 92322 Châtillon Cedex, France
3 : Helmholtz-Zentrum Geesthacht, Institute for Materials Research, D-21502 Gesthacht, Germany

* Corresponding author, Tel : 33 5 62 25 78 71, Mail : alain.couret@cemes.fr

Abstract

Thermal treatments are applied to PM TiAl alloys elaborated by Spark Plasma Sintering with the aim to improve their creep properties. Duplex microstructures are generated and characterized.

Keywords

A. titanium aluminides, based on TiAl; B. mechanical properties at high temperatures; C. powder metallurgy, including consolidation; D. microstructure (as-cast. Deformation-induced, recrystallization-induced)

1. Introduction

TiAl alloys densified by Spark Plasma Sintering (SPS) exhibit excellent mechanical properties at room temperature but a limited creep resistance in the high temperature range [1]. For instance in a $Ti_{48}Al_{48}Cr_2Nb_2$ alloy, the minimum creep rate at 700°C under 300 MPa is about 10^{-7} s^{-1}. Addition of heavy elements (Re,W) [2] or niobium [3] has failed to improve

the creep resistance because these elements segregate either into B2 precipitates or into a quasi-α_2 phase. This prevents them to reduce the mobility of dislocations in the deformed γ phase. As firstly shown by Kim [4], fine duplex microstructure formed by a mixture of lamellar and γ grains may offer a good compromise between room temperature ductility and creep strength. Previous investigations have shown that duplex microstructures are difficult to produce by classical SPS cycle, because the short processing time impedes the formation of α grains below the transus temperature. The aim of this work is to stabilize duplex microstructures and to measure their mechanical properties.

2. Experimental

Pre-alloyed gas-atomized $Ti_{48}Al_{48}Cr_2Nb_2$ (GE), $Ti_{51}Al_{47}Re_1W_1Si_{0.2}$ (G4) and $Ti_{45}Al_{46}Nb_9$ (TNB) powders, were sintered using a 2080 Sumitomo SPS equipment following procedures described elsewhere [5]. The SPS technique consists of simultaneously applying an uniaxial pressure and current pulses of high intensity to the powder enclosed in an graphite mold. This results in fast heating rates and short annealing durations (typically, 2 minutes). In the present work, either increased annealing durations of up to 30 min, or post thermal treatments were performed. The sample temperatures given in the present paper are the sample temperatures as calibrated in Ref. [5]. The samples were 36 mm in diameter and 8 mm thick. They were metallographically prepared for microstructure observations following standard procedures [1-3]. Cylindrical tensile specimens with a gauge section of 2.2 mm and a gauge length of 10 mm have been machined for room temperature straining experiments and creep tests. Tensile tests were performed at a constant strain rate of 10^{-4} s^{-1}. The creep tests were performed at 700°C under 300 MPa, under constant stress, on a machine equipped with a loading system assisted with a servo-motor having stress or deformation control.

3. Results

With the GE powder, the conditions to obtain a fine duplex microstructure have been identified. Fig. 1 shows scanning electron microscopy (SEM) micrographs in back scattered electron (BSE) mode of the microstructures obtained at 1260°C and 1285°C, for annealing durations of 2 min and 30 min. Note that for this powder, an α transus temperature between 1310°C and 1335°C was determined [5]. For the conditions 1260°C - 2 min, a double-phased microstructure (γ - α_2) is observed: the γ grains (in dark) are surrounded by small α_2 grains (in grey). Heterogeneous distribution of the α_2 grain can be emphasized. For 1285°C - 2 min, a moderate growth of γ and α_2 grains is observed. The inset shows that some α grains, but not all, have transformed into lamellar grains during cooling. For 1260°C – 30 min, a duplex microstructure formed by γ and lamellar grains is observed. The lamellar grains are larger than α_2 grains for 2 min of annealing at 1260°C, and concentrated in areas which were formerly dendrites in the powder particles, due to a local enrichment in titanium [6]. For 1285°C-30 min, the lamellar grains have grown whereas the γ grain size is nearly the same. Again, heterogeneous distribution in the lamellar and γ grains is observed.

Duplex microstructures have also been made from G4 and TNB pre-alloyed powders (Fig. 2). For the G4 alloy, heat treatments have been carried out at 1320°C for 4 h in an external furnace, starting with a two-phased sample (processed by SPS at 1285°C for 2 min). For the TNB alloy, a duplex microstructure was obtained by prolonging the SPS 1330°C temperature plateau to 30 min. The grain sizes are similar for these two microstructures, with a higher proportion of lamellar zones for the G4 alloy. As shown in the inset, very small B2 precipitates are detected at the periphery of the lamellar grains for the G4 alloy. The duplex microstructures of the TNB and G4 alloys are more homogeneous compared to the GE alloy (Fig. 1d).

Fig. 3 shows room temperature tensile and creep curves for the three duplex microstructures. Table 1 gives the mechanical data extracted from these experiments, in comparison to previous studies. At room temperature, tensile ductility is close to 1.5 % and the three alloys exhibit high yield stresses, slightly lower than those measured for double-phase microstructures. Regarding creep properties, the duplex GE alloy is not better than materials with double-phased microstructures [1]. On the contrary, the creep strength of G4 and TNB alloys is largely improved: with respect to the double-phased G4 and TNB [2,3], the minimum creep rate is reduced by nearly one order of magnitude.

4. Discussion

The creep properties of the present duplex GE alloy are only slightly better than those of double-phased GE alloys. This probably results from a high proportion of γ phase, which differentiates the microstructure not significantly (from a mechanical point of view) from a double phased one. A homogeneous duplex microstructure should exhibit better creep properties. However, Fig. 1 demonstrates that increasing temperature or time leads to the lamellar grain growth inside the former dendrites in spite of a homogenization of the microstructure. On the other hand, creep properties of G4 and TNB alloys are improved by heat treatments probably because these treatments lead to a fine microstructure and erase some heterogeneities. In the G4 alloy the volume fraction of B2 precipitates is strongly reduced, which probably contributes to the redistribution of heavy elements.

What is also remarkable for these two duplex microstructures is the improvement of the ductility with the retention of relatively high yield stresses (Tab. 1). This is particular true for the TNB alloy. This probably results from the better homogeneity of the microstructures and from erasing the dendritic structure during long thermal treatments. Because both lamellar and single-phased grains of the duplex microstructures are able to deform at room temperature by

ordinary dislocations and twinning [7,8], such duplex microstructures are expected to deform homogeneously, which should be favorable to tensile ductility.

5. Conclusions

In this work, it has been demonstrated that duplex alloys densified by Spark Plasma Sintering and containing heavy elements exhibit improved creep properties. These duplex microstructures are formed by applying long thermal treatments during or subsequent to the SPS cycle. Moreover, this high temperature strengthening is not detrimental for tensile ductility at room temperature. More generally, from a practical point of view, this work illustrates that thermal treatments can be directly applied during the Spark Plasma Sintering and thus, that external and subsequent treatments can be avoided.

Acknowledgements

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Alloys		Room Temperature (Tensile)		Creep (700°C-300MPa)		Ref
Types	Microstructures	YS(MPa)	A(%)	V(s^{-1})	Life times	
GE	Double-phased	568	2.73	2. 10^{-7}	Interrupted	[1]
G4	Double-phased	650	1.28	1. 10^{-7}	240	[2]
TNB	Double-phased	774	0.88	8. 10^{-8}	562	[3]
GE	Duplex	432	1.37	1. 10^{-7}	190	
G4	Duplex	520	1.43	3. 10^{-8}	1012	
TNB	Duplex	694	1.47	3. 10^{-8}	942	

Table 1 : Summary of the mechanical properties of TiAl alloys densified by Spark Plasma Sintering with various composition and microstructures.

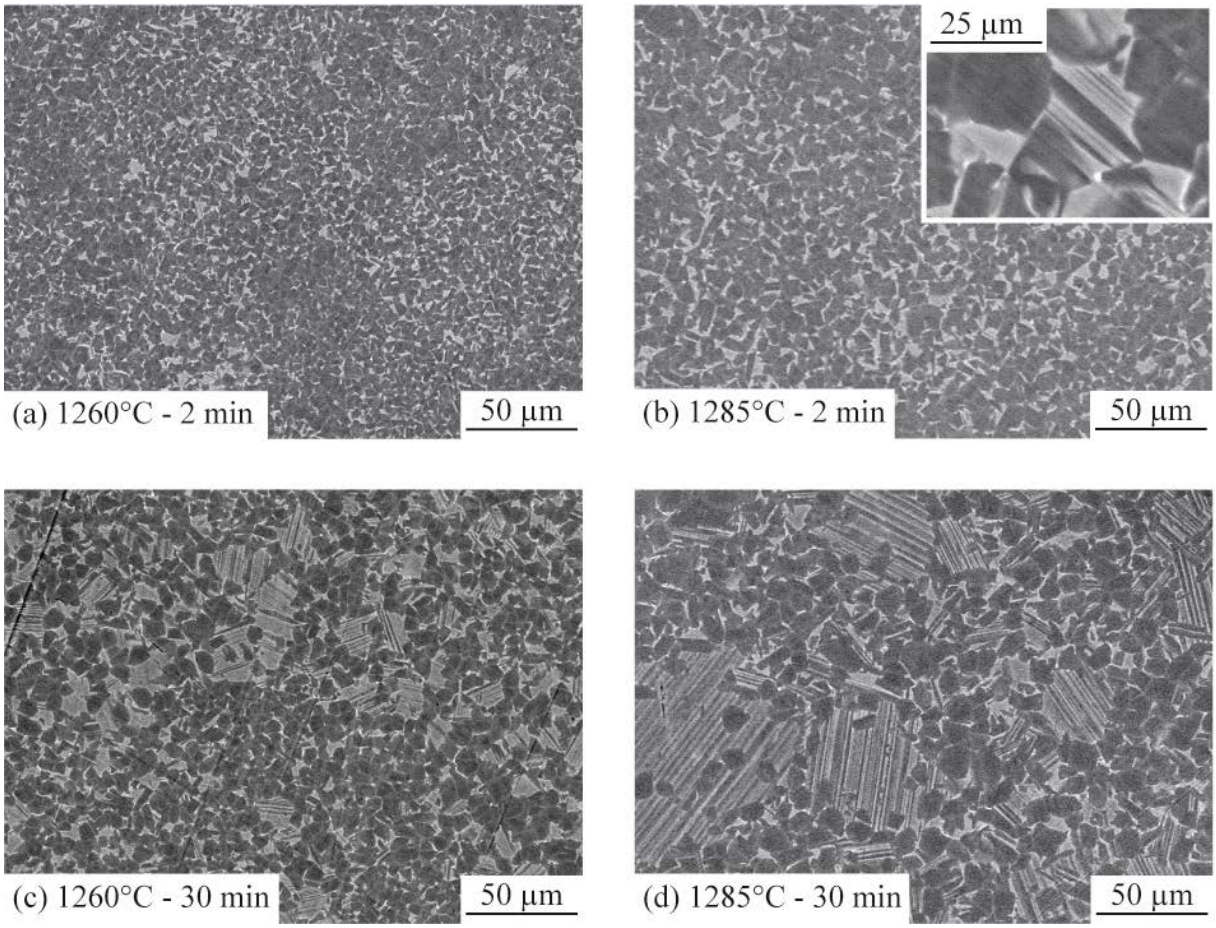

Fig. 1. Microstructures of the GE-SPS alloys processed at 1260°C and 1285°C for 2 to 30 min. Inset: higher magnification image of lamellar and α_2 grains.

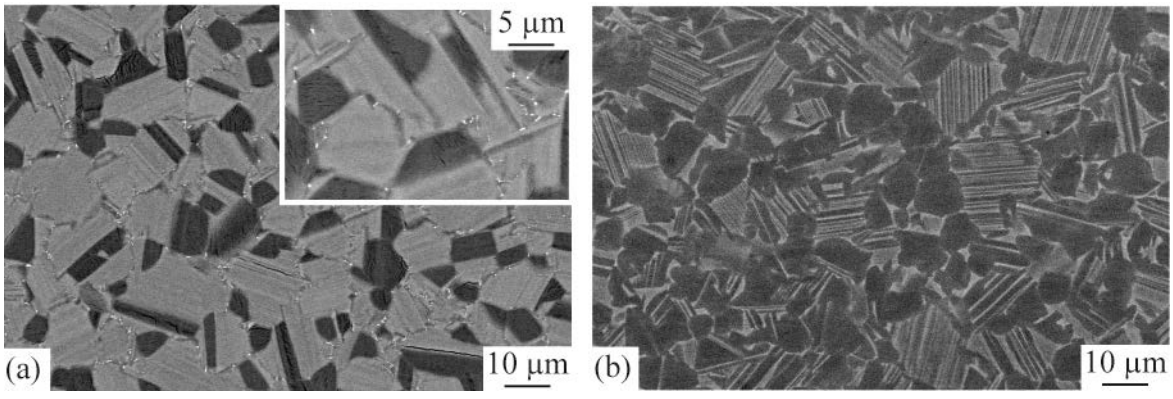

Fig. 2. Duplex microstructures of G4 and TNB alloys. (a) G4 alloy processed by SPS (1285°C, 2 min) and subsequently annealed in a furnace at 1320°C for 4 h. (b) TNB alloy annealed by SPS at 1290°C for 30 min.

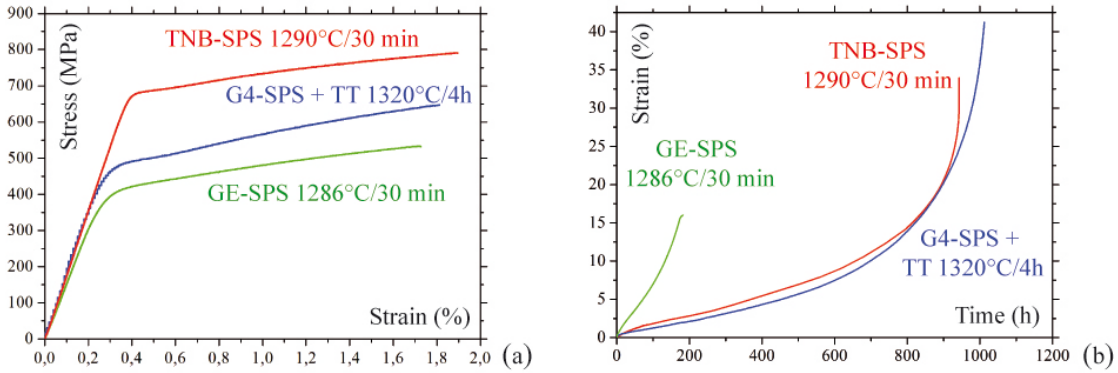

Fig. 3. Mechanical properties of duplex microstructures. (a) Room temperature tensile test curves, and (b) creep curves (700°C, 300 MPa) of the GE alloy processed by SPS at 1285°C for 30min, G4 alloy annealed in an external furnace at 1320°C for 4 h, and of the TNB alloy processed by SPS at 1290°C for 30 min.