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Principles of achieving superior superplastic properties in intermetallic alloys based on γ -TiAl + α_2 -Ti₃Al

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ABSTRACT

Keywords: Titanium aluminides Hot forging Fine grained microstructure Superplasticity Superplastic behavior of the Ti-43.7Al-4.2Nb-0.5Mo-0.2B-0.2C alloy (at. %) in a fine grained condition consisted of ultrafine grains and remnant lamellar colonies has been investigated. The fine grained condition was produced via two-stage hot forging with decreasing the forging temperature in the temperature range of T = 1200-950 °C. The produced material was used for preparation of specimens for tensile testing. The tensile tests were performed at T = 800-1000 °C and $\varepsilon' \sim 10^{-4} \cdot 10^{-3}$ s⁻¹ in air without any protection against oxidation. The tests showed incredible superplastic elongations ($\delta = 1270-2860\%$) at T = 900-1000 °C and enhanced values of the strain rate sensitivity coefficient, m > 0.3 at $\varepsilon' \sim 10^{-4} \cdot 10^{-3}$ s⁻¹. The obtained superplastic properties and microstructural examination of the tensile strained specimens suggest that the grain boundary sliding was the predominant deformation mechanism during superplastic flow. Superior superplastic properties were attained owing to unusually slow dynamic grain growth during superplastic flow. This was supported by a high content of the α_2 -Ti₃Al phase, high alloying with niobium and dynamic recrystallization/globularization during superplastic flow leading to transformation of remnant lamellar colonies into the fine grains. The activation energy was defined as $Q \approx 310 \text{ kJ/mol}$ suggesting that the predominant deformation mechanism during superplastic flow was grain boundary sliding controlled by volume diffusion of aluminum and titanium in γ -TiAl and α_2 -Ti₃Al. On basis of the performed study, the principles of achieving superior superplastic properties ($\delta > 1000\%$) in intermetallic alloys based on γ -TiAl + α_2 -Ti₃Al have been proposed.

1. Introduction

It is now 30 years since the first paper devoted to superplasticity of intermetallic alloys based on γ -TiAl + α_2 -Ti₃Al (hereafter $\gamma + \alpha_2$ alloys) was issued [1]. Later a number of investigations on superplasticity in γ -TiAl + α_2 -Ti₃Al alloys has been carried out and published [2–35]. It has been believed that superplastic properties are improved: i) with refining the microstructure down to $d \sim 0.1 \,\mu\text{m}$, ii) with increasing the α_2 -Ti₃Al phase content, iii) at transition from "conventional" peritectic alloys to β -solidifying ones, which are chemically more homogeneous, iv) with appearance of the $\beta(B2)$ phase, which is regarded to be favorable for superplasticity. As in conventional alloys, superplastic elongations in intermetallic $\gamma + \alpha_2$ alloys are limited by dynamic grain growth, strain localization and porosity and typically do not exceed 700%. However superplastic behavior of $\gamma + \alpha_2$ alloys continues to surprise. It has been recently revealed extraordinary superplastic properties in the ultrafine grained alloy with the nominal composition Ti-45Al-8Nb-0.2C [36]. In the alloy, the α_2 content was defined only as about 3 vol% and the $\beta(B2)$ phase was not detected at all. Nevertheless, the elongations as high as $\delta = 1003-1342\%$ were reached at T = 900-1000 °C that seems to be paradoxical taking into account that single-phase materials, particularly pure metals, are incapable of superplasticity because of fast dynamic grain growth during straining [37,38]. The superplastic behavior of the Ti-45Al-8Nb-0.2C alloy was explained in terms of the ultrafine grained microstructure facilitating grain boundary sliding and the higher niobium content, which reduced diffusivity and dynamic grain growth during superplastic flow [36]. To further restrict the dynamic grain growth during superplastic flow, it makes sense to increase the second phase content. To realize this new "old" idea, the α_2 phase is well suited, whereas the $\beta(B2)$ phase being very soft at elevated temperatures can provoke the strain localization within this phase impeding achievement of a uniform fine grained condition during hot working and restricting superplastic elongations. Indeed, highest elongations attained in the $\gamma + \beta(B2) + \alpha_2$ alloys with stable or metastable $\beta(B2)$ phase both in cast and wrought conditions were always less than 700% [13,18,25,28,30,32-35]. Reasoning from the aforesaid, superplastic behavior seems to be interesting to study for a $\gamma + \alpha_2$ alloy containing a large amount of the α_2 phase and free or near free of the

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Fig. 1. BSE images of the Ti-43.7Al-4.2Nb-0.5Mo-0.2B-0.2C alloy: (a) the cast condition; (b) after hot forging and ageing; (c,d) after superplastic deformation at T = 1000 °C and $\varepsilon' = 8.3 \times 10^{-4}$ s⁻¹ ($\delta = 2860\%$), (c) grip area and (d) gage area of the specimen near the fracture zone.

 $\beta(B2)$ phase. So called β -solidifying TNM $\gamma + \alpha_2$ based alloys typically alloyed with 4–5 at.% Nb, 1 at.% Mo and 0.1–0.2 at.% B are well suited to test this idea.

Thus, the present work was aimed to study superplastic behavior of one of the advanced TNM $\gamma + \alpha_2$ alloys in a fine grained condition. A lower content of β -stabilizing molybdenum and an addition of carbon, which is known as a strong α -stabilizing element, were used in contrast to the most studied Ti-(43–43.5)Al-4Nb-1Mo-0.1B (at. %) alloy [39,40] to provide a higher content of the α_2 phase and a lower content of the $\beta(B2)$ phase. To produce the fine grained condition, two-stage hot forging was applied. Based on the study and earlier performed works, the principles of achieving superior superplastic properties in γ -TiAl $+\alpha_2$ -Ti_3Al based alloys are to be formulated.

2. Material and experimental

The starting material with the nominal composition of Ti-43.7Al-4.2Nb-0.5Mo-0.2B-0.2C (at. %) was produced by vacuum arc remelting and supplied from GfE Metalle und Materialien, Germany as as-cast ingots of Ø95 × 180 mm. The as-cast material was annealed at T = 1275 °C ($\tau = 0.5$ h) followed by ageing at T = 900 °C ($\tau = 3$ h) in order to equilibrate the phase composition and dissolve the metastable β (B2) phase. The heat treated condition is indicated in the text as the cast condition.

The hot forging was performed in two stages. The first stage was conducted under quasi-isothermal conditions in the $\alpha + \beta(B2) + \gamma$ temperature field. To do it, die tool was preheated up to T = 950 °C and the ingot was canned using low-carbon steel in order to provide quasi-isothermal conditions and protection from oxidation during forging and then heated up to T = 1200 °C. The first hot forging was fulfilled at a strain rate of $\varepsilon' = 10^{-2} \cdot 10^{-1} \text{ s}^{-1}$. After the first-stage, the workpiece was decanned and subjected to intermediate annealing. The second stage was conducted under isothermal conditions in the $\alpha_2 + \beta(B2) + \gamma$ phase field at T = 950 °C, $\varepsilon' \sim 10^{-3} \text{ s}^{-1}$. At first, it was made in the same forging direction. Thereafter the obtained forging was cut into workpieces, which were subjected to 3D forging under isothermal conditions at T = 950 °C, $\varepsilon' \sim 10^{-3} \text{ s}^{-1}$ followed by ageing at T = 900 °C (2 h) and furnace cooling. The 3D forging procedure

included alternate forging in three directions that finally led to sound forgings free of any cracks with an approximate size of \emptyset 50 × 14 mm. The summed true strain imparted to the material during hot forging can be evaluated as follows: $e = \Sigma \ln(H_{in}/H_{fin})$ (1), where H_{in} and H_{fin} are the heights of the workpiece before and after each forging step. According to (1), the summed strain imparted during two-stage forging procedure was $e \approx 4$. The forged material was further used for microstructural examination and preparation of specimens for tensile testing.

Microstructural examination was carried out using scanning electron microscopy in backscattering electron mode (SEM, BSE). Before SEM studying, the specimen surfaces were polished. The specimens for SEM examination were prepared from the grip area and near the fracture zone of the tensile tested specimens. BSE images were used to define the volume fractions of remnant lamellar colonies and the α_2 phase, as well the mean grain size, *d* in the grips and the gauge areas of tensile tested specimens. The alloy composition was measured by energy dispersive X-ray (EDX) analysis system.

Flat specimens with a gauge size of $10 \times 5 \times 2 \text{ mm}^3$ were tensile tested at T = 800-1000 °C with an initial strain rate of $\varepsilon' = 8.3 \times 10^{-4}$ and $8.3 \times 10^{-3} \text{ s}^{-1}$. The tensile tests were performed in air without any protection against oxidation. An elongation to rupture, δ , and true stress-true strain curves σ -e were determined. The strain rate sensitivity coefficient, *m*, was defined at different strain rates by changing the strain rate employed. To plot the dependence of $\ln(\sigma)$ on 1/T, the σ_{30} values corresponding to 30% of elongation were taken into consideration.

3. Results and discussion

3.1. Effect of processing on microstructure

EDX analysis confirmed the alloy composition with the exception of the aluminum content, which was found to be slightly lower in contrast to the nominal composition, about 43.2 at. %. Fig. 1a and b represents BSE images of the Ti-43.7Al-4.2Nb-0.5Mo-0.2B-0.2C alloy in the cast and forged conditions. The cast material had near fully lamellar structure with a mean colony size of $d = 63 \pm 5 \mu m$, the volume fraction of the β (B2) phase was defined as about 1%. In the microstructure there

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