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TiAl-based high temperature materials made from extremely deformed elemental powders

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Materials based on the intermetallic compound TiAl have been made from elemental powders which were extremely deformed through repeated cold-extrusion or through combination of cold-extrusion and cold-rolling. The microstructure is getting finer with increasing deformation. After HIP at 1350°C under a pressure of 200 MPa for 4 hours without encapsulation, the repeatedly extruded material showed a porosity of about 2%, smaller than the porosity of simply extruded material (> 6%). Two-step sintering experiments carried out on the repeatedly extruded material showed that pores formed during pressureless sintering can be almost fully suppressed by an additional HIP treatment. On the contrary, the relatively low porosity HIP'ed material becomes more porous after a post pressureless sintering. In addition, TiAl matrix composites have been successfully prepared by pressing multilayered rolled sheets, sandwiched with Al₂O₃ fibres or Mo foils, at high temperatures.

1. Introduction

Titanium aluminides as potential materials for high temperature applications are mostly produced using ingot metallurgy. However, the poor workability of titanium aluminides directs the attention to the production route of reactive powder metallurgy. This route includes following essential steps: mixing of elemental powders with desired composition, precompaction, cold-extrusion, and reactive sintering. During the reactive sintering, an external pressure is normally applied in order to suppress pore formation caused by different diffusivities of Ti- and Al-atome. This can be realized e.g. by hot isostatical pressing (HIP) as-extruded materials. With this process, the poor workability of titanium aluminides is avoided and nearly fully dense materials can be prepared by applying encapsulating techniques. Since encapsulation is normally very expensive, HIP without encapsulation is desirable in view of economy. Earlier work [1] showed that a large deformation of powders prior to HIPing is beneficial not only to increasing materials density but also to accelerating formation of titanium aluminides. A largely refined microstructure of the as-extruded material leads also to a reduction of pore size as well as porosity during reactive sintering [1]. The open porosity must be also reduced, so that the expensive encapsulation may be omitted. It was therefore attempted in this study to carry out HIP treatment of the extruded materials without encapsulation. Two-step sintering experiments were also conducted to see whether the more expensive HIP time can be shortened. In addition, TiAl matrix composites were successfully prepared by HIPing or HPing (hot pressing) multilayered as-rolled sheets, sandwiched with Al₂O₃ fibres or Mo foils.

2. Experimental procedure

Elemental powders with a size $< 150 \mu\text{m}$ were mixed in air using a tumbling mixer. The composition was Ti-34wt.%Al, i.e., Ti-48at.%Al. The powders used were water-atomized 99.5% pure aluminium and titanium sponge with the same purity. Green compacts were prepared at room temperature in an uniaxial pressing machine. Compacted billets were extruded at room temperature to bars with either a round cross-section of 12 mm diameter (extrusion ratio ≈ 17) or a 10 mm x 20 mm rectangular one (extrusion ratio ≈ 10). Afterwards, the round bars were further extruded to give a total extrusion ratio of 350, and those with a rectangular cross-section were cold-rolled to 0.5 mm and 0.1 mm thick sheets, with the rolling direction being perpendicular to the extrusion direction.

Both the simply (with extrusion ratio of 17) and repeatedly (with extrusion ratio of 350) extruded materials were hot isostatically pressed at 1350°C under a pressure of 200 MPa for 4 hours without encapsulation. Since the cost for HIP treatment increases with increasing HIP time, it is of practical importance to reduce the HIP time. However, this is limited because of the time required to complete the reaction between elemental Ti and Al to form the intermetallics TiAl and Ti_3Al . In addition, the process of pore shrinkage during HIP is also time-dependent (creep process). In this respect, a two-step sintering, i. e. a combination of the relatively cheaper pressureless sintering and the expensive HIP treatment, seems to be reasonable. Therefore, two variations were carried out on the repeatedly extruded material:

Scheme 1: pressureless sintering ($1350^\circ\text{C}/6 \text{ h}$) + HIP ($1350^\circ\text{C}/4 \text{ h}/200 \text{ MPa}$)

Scheme 2: HIP ($1350^\circ\text{C}/4 \text{ h}/200 \text{ MPa}$) + pressureless sintering ($1350^\circ\text{C}/6 \text{ h}$)

In order to prepare TiAl matrix composites with strength and ductile reinforcements, HIPing and HPing of multilayered as-rolled sheets, sandwiched with Al_2O_3 fibres (as strength reinforcements) and Mo foils (as ductile reinforcements), respectively, were conducted. The following conditions were applied:

HIP: $1350^\circ\text{C}/2 \text{ h}/200 \text{ MPa}$

HP: $1250^\circ\text{C}/2 \text{ h}/20 \text{ MPa}$

3. Results

3.1. Microstructure of the as-extruded and as-rolled materials

Figure 1 shows the as-extruded microstructures of extrusion ratios of 17 and 350 as well as the as-rolled microstructures of 0.5 mm and 0.1 mm thick sheets. It can be seen that the microstructure with extrusion ratio of 350 is much finer than that with extrusion ratio of 17. Also the as-rolled microstructure is getting finer with reducing sheet thickness. Both Ti and Al powders are elongated in the extrusion or rolling direction to fibres. Almost no pores can be detected. The as-extruded as well as the as-rolled materials are strongly textured because of the alignment of both Al- and Ti-fibres in extrusion direction. The reaction of elemental Ti and Al to intermetallics did not take place during extrusion, since only intensity peaks of pure Ti and Al were measured by x-ray diffraction analysis [2]. These materials can be therefore easily machined or reformed to different shapes. This is of practical importance, since the presence of titanium aluminides would make the manufacture much more difficult.

3.2. Porosity after HIP-treatment and two-step sinterings

After HIPing at 1350°C under a pressure of 200 MPa for 4 hours without encapsulation, a porosity of about 2.1% was measured on the repeatedly extruded material, lower than the porosity ($> 6\%$) of the simply extruded material (Fig. 2).

The microstructures after both two-step sinterings are shown in Figs. 3a and 3b, respectively. It

is visible that the treatment scheme does influence the final porosity. The porosity after treatment of the scheme 1 is only 0.7%, much smaller than that for the scheme 2 (5.0%). The pores formed during pressureless sintering 1350°C/6 h (about 2% [1]) have been almost fully suppressed by the additional HIP treatment (Fig. 3a). On the contrary, the relatively low density HIP'ed material (Fig. 2a) became more porous after the post pressureless sintering at 1350 °C/6 h (Fig. 3b).

Fig. 1: As-extruded microstructures of extrusion ratios of 17 (a) and 350 (b) as well as the as-rolled microstructures of 0.5 mm (c) and 0.1 mm (d) thick sheets

Fig. 2: Pores formed during HIP (1350 °C/4 h/200 MPa) in specimens extruded with extrusion ratios of 17 (a) and 350 (b), respectively

Fig. 3: Pores after two-step sinterings; (a) scheme 1 and (b) scheme 2

3.3. Microstructure of the composites

Figure 4 shows the photographs taken from the prepared composites with Al_2O_3 fibres (Fig. 4a) and Mo foils (Fig. 4b). For TiAl+ Al_2O_3 composite (Fig. 4a), 0.1 mm thick sheets (Fig. 1d) were used, whereas use of 0.5 mm thick sheets (Fig. 1c) and 0.1 mm thick Mo foils was made for TiAl+Mo composite (Fig. 4b). A duplex microstructure with lamellar TiAl/ Ti_3Al grains in equiaxed TiAl-phase is seen in the TiAl matrices of both composites, with the lamellar volume fraction in TiAl+ Al_2O_3 composite being higher than in TiAl+Mo composite [3]. Part of the Al_2O_3 fibres in Fig. 4a were dissolved due to the high temperature of 1350 °C. The reaction zone between TiAl and Mo exhibits a thickness of about 10-20 μm (Fig. 4b).

4. Conclusions

The results shown above can be summarized as below:

- a. With increasing deformation, microstructure of consolidated elemental Ti and Al powder mixture becomes finer. This exerts a strong influence on the porosity formed during reaction HIP process without encapsulation. The finer the microstructure, the lower the porosity.
- b. To reduce the more expensive HIP time, a combination of pressureless sintering and HIP can be done. The treatment sequence does affect the final porosity. For the repeatedly extruded material in this study, additional HIP after pressureless sintering leads to a porosity reduction. On the contrary, an increase of porosity is observed after post pressureless sintering of the HIP'ed material, probably due to Ostwald ripening [2].
- c. TiAl+ Al_2O_3 and TiAl+Mo composites can be prepared by HIPing or HPing multilayered as-rolled sheets, sandwiched with Al_2O_3 fibres (as strength reinforcements) and Mo foils (as ductile reinforcements), respectively. To limit the undesired reactions between TiAl matrix and the reinforcements, low temperatures should be applied for HIP or HP. But, the time required to complete the TiAl formation will be longer with decreasing temperature. An appropriate temperature range is still to be found out through more experiments in the future.

Fig. 4: Prepared composites; (a) TiAl+ Al_2O_3 and (b) TiAl+Mo

5. Literature

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