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# Microstructure and properties of Ti–45Al–5V intermetallic alloy

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An alloy of the chemical composition Ti-45Al-5V (at. %) was synthesized by mechanical alloying in a Szegvari-type attritor from elemental powders of high purity. The powders were further consolidated by hot isostatic pressing and hot isostatic extrusion. The resulting material was composed of a mixture of TiAl- and $Ti_3Al$-based phases. However, no lamellar microstructure, typical of such alloys, was observed. The alloys exhibited exceptionally high yield strength, together with satisfactory ductility and fracture toughness. The high strength was unequivocally due to grain refinement and the presence of oxide dispersoid.

Key words: *intermetallics; mechanical alloying; TiAl; $Ti_3Al$*

## 1. Introduction

TiAl-based alloys are promising materials for high temperature applications, especially for industrial and aviation turbines as well as for the aerospace and automobile industries. Their advantageous properties include low density, high melting point, good oxidation and burn resistance, completed with high yield strength and high elastic modulus, which is retained at elevated temperatures. The disadvantages of the TiAl-based intermetallic materials, consequently limiting their application, are brittleness at room temperature and a rapid loss of mechanical properties at temperatures above 700 °C [1].

Various approaches have been attempted to improve the properties of TiAl-based alloys. For example, greater ductility and fracture toughness were obtained by modifying the chemical composition, by alloying with substiutional elements such as vanadium [2]. Another alternative for enhancing the properties of TiAl-based alloys is the application of novel processing methods, by achieving accurate control of size and

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distribution of constituent phases (in particular refinement of the lamellar microstructure) as well as by refining the grain size [3].

This research was aimed at processing TiAl-based materials, modified by the addition of small amounts of vanadium. Mechanical alloying (MA) was selected as the processing route. MA has the advantage of enabling grain size refinement down as far as the nanoscale; another advantage is the possible generation of non-equilibrium and amorphous phases. Also, the controlled oxygen level in the milling chamber produces oxide dispersoid, which is believed to contribute to the retention of strength and creep resistance at elevated temperatures, regardless of how fine-grained the microstructure is.

## 2. Experimental

An alloy with the composition Ti–45Al-5V (at. %) was synthesized in a Szegvari laboratory attritor mill. The excess titanium, with respect to stoichiometry in the alloy, was included in order to ensure that the alloy would exhibit a two-phase microstructure (near γ). The alloy was fabricated by mechanical milling of high purity elemental powders. All the powder particles had an average diameter lower than 44 μm (325 mesh). Mechanical alloying was carried out in an argon atmosphere: the controlled oxygen level was reduced to about 3–5 ppm. The controlled amount of oxygen was believed to produce oxide dispersoid, and consequently it was expected to improve the strength of the final material. A slight positive pressure (ca. 20 kPa) was maintained inside the mill in order to prevent air entering the mill during the milling process. The milling was carried out in a 1400 $cm^3$ sealed stainless steel tank with a 3.63 kg of 4.76 mm (3/16") stainless steel balls. The ball to powder ratio (by weight) was about 11:1. The first stage of milling (2 h) was carried out at cryogenic temperatures, using liquid nitrogen as a coolant. The liquid nitrogen prevents relatively ductile powder particles from agglomerating on the tank walls, shaft arms and balls, and also promotes the initial cold fracture. During the next milling stages, water cooling was applied. The powder was sampled periodically (after 2, 10, 50 and 100 h) during the milling, allowing characterization of the powder morphology and the progress of the mechanical alloying process. The amount of powder produced in one run of the mill was usually 200–300 g. The changes in the particle morphology during milling were examined by scanning electron microscopy. X-ray diffraction (XRD) was used for evaluation of the lattice parameters, lattice strains and the X-ray crystallite sizes.

The powders collected at the end of milling were sieved through a 44 μm mesh and consolidated by hot isostatic pressing (HIP) at 1100 °C and 1200 °C under 1.4 GPa for 14 h. An additional sample was compacted by hot isostatic extrusion (HIE) at 1000 °C with reduction 4:1 (true strain about 1.4) and at a strain rate of $10^2$ $s^{-1}$. All powders were degassed before consolidation. The samples after both HIP and HIE were in the form of cylinders 10 mm in diameter. The compacted materials were subjected to XRD analysis, light microscopy, transmission and scanning electron micros-

copy (TEM and SEM) as well as mechanical tests. X-ray crystallite size and the lattice strain were determined from peak broadening according to the method proposed by Williamson and Hall [4]. The electron microscopy investigation was supplemented by chemical analysis performed by energy dispersive spectroscopy (EDS) – both in SEM and TEM. Thin foils were prepared by the ion milling method.

Mechanical tests comprised hardness and compression tests at room temperature, determination of elastic constants and evaluation of fracture toughness $K_{Ic}$. For this purpose, samples in the shape of parallelepipeds ($2.0\times2.8\times5.0$ mm$^3$) were cut out from the central part of the consolidated samples. The non-square base was selected intentionally since, prior to mechanical tests, the elastic constants were evaluated on the samples using a resonant ultrasound spectroscopic method. The compression tests were carried out at a strain rate of $3\times10^{-3}$ s$^{-1}$. The assessment of fracture toughness $K_{Ic}$ was performed from microcracks propagating from Vickers indentation corners on the prepolished sample surfaces. For this purpose, the Palmquist [5] model of microcrack formation and the mathematical analysis proposed by Niihara et al. [6] were adopted.

## 3. Results and discussion

The mechanical alloying process proceeded in a manner typical for milling ductile powders [7]. The powder particle morphology, after selected milling times, is shown in Fig. 1.

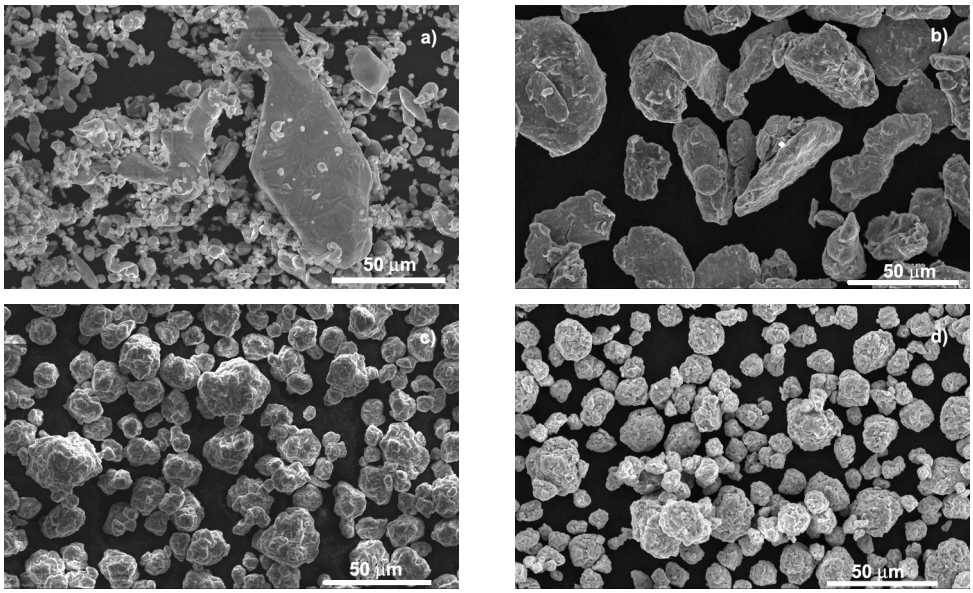


Fig. 1. SEM image of powder particles after: a) 0 h, b) 2 h, c), 50 h and d) 100 h of milling

Initially, an increase in the particle size, and their flattening was observed. This was due to the predominance of welding over fracturing occurring in relatively soft particles. Many particles had sizes approaching 500 μm, though considerable size variation was observed The particle shapes became more spheroidal with the milling time. After 50 h of milling, the particles became much finer, indicating the predominance of fracturing over welding, but rough particle surface showed that welding was still in progress (Fig. 1). 100 h of milling produced a uniform powder with almost spherical particles having smooth surfaces. The average size of a powder particle (after sieving through a 45 μm mesh) was about 10 μm. The chemical composition of individual powder particles, measured by EDS, was uniform throughout the particle volume and corresponded to the starting composition of the powder blend.

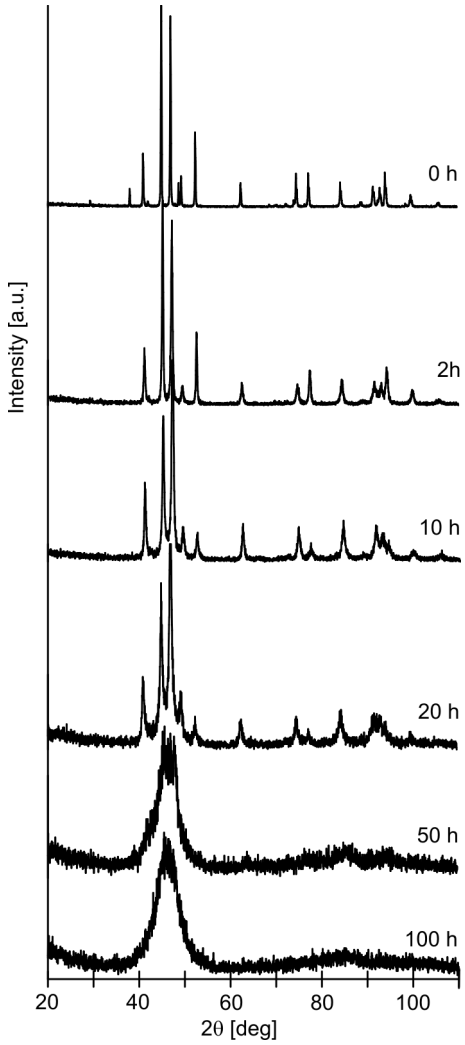


Fig. 2. XRD patterns of milled powders showing progress of mechanical alloying

X-ray diffractograms revealed clear dependences on the milling time (Fig. 2). In general, the powder behaviour during milling was typical of the mechanical alloying when solid solutions are formed [7]. The peaks became broader, their intensities decreased and most of them finally vanished as the mechanical alloying process progressed. A significant broadening of peaks suggested the formation of an amorphous phase, fine crystallite grains and/or high density of defects. The change in X-ray crystallite sizes and lattice strain for all alloys occurred in a typical manner, i.e. the crystallite sizes decreased and lattice strain increased with the milling time (Table 1).

Table 1. Crystallite sizes and lattice strains for Ti peaks

| Milling time, h | 0 | 2 | 10 | 50 | 100 |
|---|---|---|---|---|---|
| Crystallite size, nm | 819 | 215 | 165 | 31 | 18 |
| Lattice strain, % | 0.11 | 0.32 | 0.49 | 3.48 | 5.83 |

The peak positions changed a little with the milling time, i.e. only a slight shift to higher values of $2\theta$ was observed. This indicated a little alteration of the lattice parameters of the processed alloy with the milling time. The direction of this shift was expected, since atom diameters of dissolved elements in Ti (Al, V) are smaller than the diameter of Ti atoms. Minor differences exist between Ti and Al atom radii (145 pm vs. 143 pm) and these two elements are basic constituents of the alloy. That is why such a small shift in peak positions occurred in the X-ray diffractograms. A broad peak at a $2\theta$ position around 45° after 100 h of milling can be associated with reflections belonging to either pure alloy constituents (such as Al, Ti), compound TiAl or the formation of an amorphous phase. Similar effects were observed by Dutkiewicz et al. in mechanically alloyed TiAl–V alloy [8]. The XRD patterns show that vanadium dissolves easily in titanium or turns into an amorphous form.

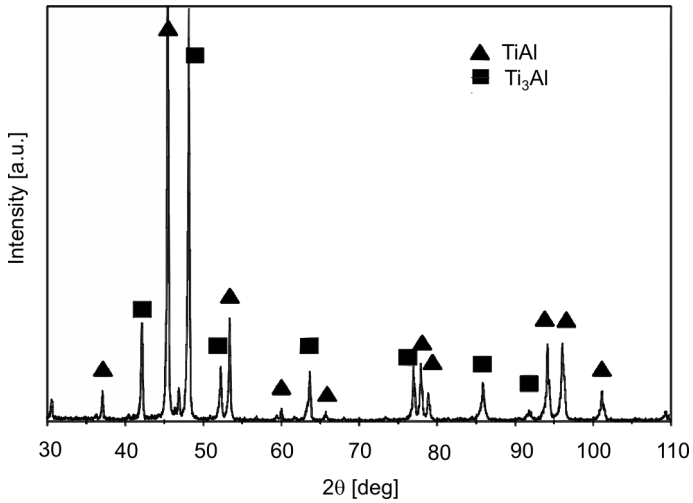


Fig. 3. Typical XRD pattern from the consolidated material; HIP at 1100 °C

The consolidation of mechanically alloyed powders produced solids of the required quality. XRD patterns from all consolidated materials were similar and showed the presence of TiAl and $Ti_3Al$-based phases (Fig. 3). An unexpected result was, however, a relatively large width of the diffraction peaks. This might have happened due to a probable presence of a fine grained microstructure and stresses arising from different coefficients of thermal expansion of particular phases.

Optical microscopy allowed the distribution of large oxide particles to be revealed. The distribution was fairly uniform throughout each HIP and HIE sample. A typical microstructure is presented in Fig. 4.

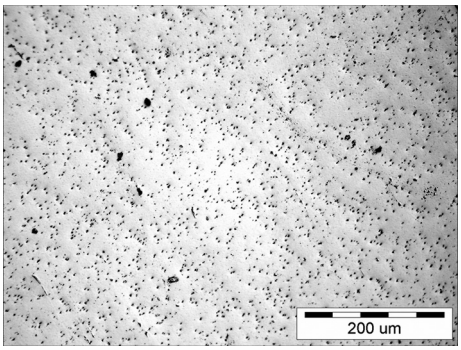


Fig. 4. Optical microstructure of the alloy consolidation by HIP at 1100 °C; not etched sample

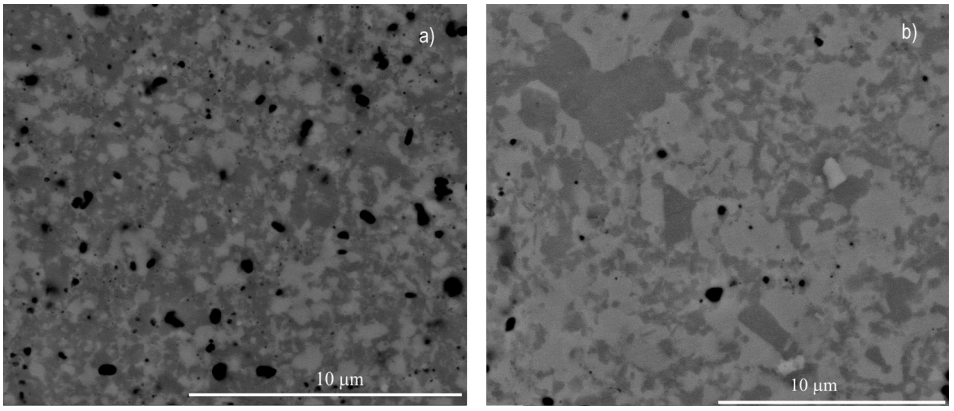


Fig. 5. Typical microstructures of the alloy consolidated by HIE (a) and HIP at 1100 °C (b); SEM BSE, contrast Z

The phases detected by X-ray analysis were also revealed in SEM images formed by backscattered electrons with utilization of Z contrast (Fig. 5). Such images are particularly useful in determining the size and distribution of constituent phases. The black particles in Fig. 5 contained mainly aluminum and oxygen (with at. % ratio 2/3) and thus were identified as $Al_2O_3$. SEM investigation confirmed that the distribution of oxides in all samples was more or less uniform. Beside oxides, the microstructure was composed of areas having different degree of grayness: brighter areas contained more Ti than Al, while in the darker ones the Ti and Al contents were similar. Taking

into account X-ray phase analysis, the brighter and darker areas were identified as phases based on $Ti_3Al$ and TiAl compounds, respectively. The interesting discovery in the present research was that the produced alloys did not exhibit a lamellar microstructure, which is typical of the two-phase γ-TiAl based alloys having similar chemical compositions but synthesized by other methods [1].

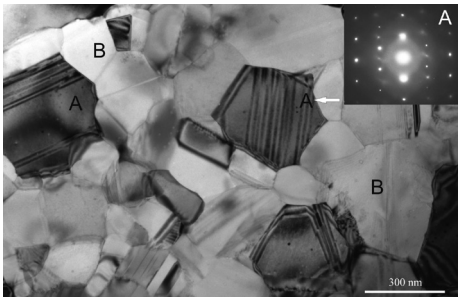


Fig. 6. TEM micrograph of the alloy consolidated by HIP at 1100 °C

A typical microstructure of the synthesized alloys, revealed by TEM, is shown in Fig. 6. TEM investigation, along with selected area diffraction pattern analysis, confirmed that the examined alloys consisted of a mixture of $Ti_3Al$-based and TiAl-based phases, regardless of the consolidation route and temperature. The two phases could be easily recognized by numerous stacking faults which occurred in the $Ti_3Al$-based as opposite to the TiAl-based phase. Both phases were built of colonies of small grains (<1 μm), however, the chemical compositions varied in particular grains belonging to the same phase. The content of V was partitioned evenly into both phases and its content was near the targeted value of 5 at. %. This unequivocally shows that vanadium diffuses easily into TiAl and $Ti_3Al$ solid solutions. The Ti:Al atomic ratio in the TiAl based phase was close to 1. On the other hand, the chemical composition of the $Ti_3Al$ based phase was far from stoichiometric, with Ti content ranging from 57 to 67 at. %. The presence of the Al-rich $Ti_3Al$ based phase is detrimental to the mechanical properties of these alloys since it is considered as a primary source of brittleness at room temperature [9]. Density and elastic constants of consolidated materials, together with their mechanical properties, are listed in Table 2.

Table 2. Physical and mechanical properties of the MA Ti-45Al-5V alloy at room temperature

| Property | Consolidation method | | |
|---|---|---|---|
| | HIE 1000 °C | HIP 1100 °C | HIP 1200 °C |
| Density, g/cm$^3$ | 3.99 | 3.99 | 4.00 |
| Young modulus, GPa | 180 | 175 | 179 |
| Poisson ratio | 0.20 | 0.26 | 0.26 |
| Yield strength, MPa | 2500 | 2500 | 1800 |
| Hardness, HV | 590 | 630 | 613 |
| $K_{Ic}$, MPa·m$^{0.5}$ | 4 | 6 | no microcraks at indent corners |

The yield strengths of all examined materials were significantly higher than in other γ-TiAl alloys having similar chemical compositions, which is usually in the range of 360–450 MPa, depending on the alloy microstructure [1]. The higher strength of the material described in the present paper was unequivocally a result of grain refinement and the presence of oxide dispersoid. It was found that the physical and mechanical properties depended to some degree on the consolidation method. Higher ductility was observed for HIP-ed materials which increased with the consolidation temperature (Fig. 7). However, the ductility was not accompanied by any considerable strain hardening. Samples consolidated at 1000 °C and 1100 °C showed an exceptionally high yield stress of about 2500 MPa. Samples compacted at 1200 °C exhibited a lower yield stress but their ductility was much better.

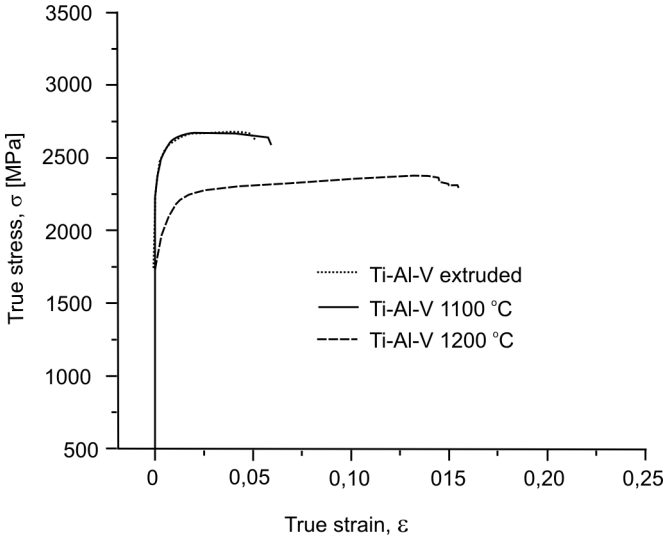


Fig. 7. Stress–strain curves for the alloys deformed at room temperature

A similarly high level of strength in TiAl-based alloys was also reported by Bohn et al. [10] for a Ti-48Al alloy with a grain size of around 130 nm, and by Calderon et al. [11] for a nanocrystalline Ti–46Al–4Cr alloy, also produced by mechanical milling but consolidated by spark plasma sintering – the method which makes it possible to obtain a nanostructure in bulk materials, due to short processing times. It is worth noting that the present Ti–45Al–5V alloy, consolidated by HIP or HIE, shows extremely high flow stresses without having a lamellar microstructure, which is usually considered for achieving the highest strength among all γ-type alloys [1]. The high yield strengths obtained in the present research are certainly associated with the alloying with vanadium, the fine grain size as well as the presence of oxide dispersoid.

The hardness of the examined materials varied with the method of consolidation and cannot be correlated with yield strength. A similar conclusion may be drawn about the fracture toughness, which depends mainly on the consolidation temperature.

## 4. Conclusions

The applied processing route (i.e., mechanical alloying followed by hot isostatic pressing or hot isostatic extrusion of powders) can be successfully used for the production of TiAl-based alloys having exceptionally high yield strength, together with some ductility and satisfactory fracture toughness.

The high strength of the produced intermetallic alloy was unequivocally a result of grain size refinement and the presence of oxide dispersoid.

The produced alloys are composed mostly of TiAl- and $Ti_3Al$-based phases, however no lamellar microstructure, typical for TiAl-based intermetallic, was observed.

Vanadium partitioned equally into both TiAl- and $Ti_3Al$-based phases.

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