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# STRUCTURE AND MECHANICAL PROPERTIES OF BALL MILLED TiAl-Cr INTERMETALLICS CONSOLIDATED BY HOT PRESSING AND PULSE PLASMA SINTERING

**W. Maziarz[1], A. Michalski[2], P. Kurtyka[3] and J. Dutkiewicz[1,3]**

[1]Institute of Metallurgy and Materials Science of the Polish Academy of Sciences, Kraków, Reymonta 25, Poland
[2]Warsaw University of Technology, Department of Materials Science and Engineering, Warsaw, Wołoska 141, Poland
[3]Pedagogical Academy, Kraków, Podchorążych 2, Poland

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**Abstract.** The TiAl-Cr alloys compositions were chosen from the γ-phase stability region up to maximum 5 at.% chromium. The elemental powders were initially blended to desired composition of Ti-49Al-49Cr-2 and Ti-48Al-47Cr-5 (numbers indicate at.%). The ball milling process was performed in a high energy planetary mill Fritsch Pulverisette P5/4. Formation of mixture of amorphous and nanocrystaline Cr(Ti, Al) solid solutions was observed after 40 hours of milling. Two consolidation methods of milled powders were applied: hot pressing (HP) at 1273K at 35 MPa under argon flow and pulse plasma sintering (PPS). The conditions of the latter method were optimized for pressure and voltage by application of temperature and shrinkage measurements during the process. The micro-hardness of consolidated samples was about 860 $HV_1$ and only small differences were observed between samples obtained by both methods. The mean density of pulse plasma sintered compact was about 98% of the theoretical value while hot pressed samples had lower density close to 95% of the theoretical one. The TEM observation of consolidated samples revealed that pulse plasma sintered samples have a grain size in the range of tens of nanometers while the grains in the hot pressed samples are larger, of size below 1 micron.
The compression tests performed in the temperature range between RT and 1073K with deformation rate of $4 \cdot 10^{-4} s^{-1}$ demonstrated higher ductility and strength of PPS samples at room temperature, due to a different level of densification and grains size.

## 1. INTRODUCTION

Alloys based on the γ-TiAl intermetallic exhibit high strength and oxidation resistance at elevated temperatures. These features in combination with their low density allow higher working temperatures and lead to improved operation efficiency and fuel saving in advanced transportation systems [1-4]. However, they exhibit poor ambient temperature ductility and fracture toughness which has limited widespread applications of these materials [2]. Microstructural refinement via ternary additions and far-from-equilibrium processing methods can improve these properties [1,2]. The Mechanical Alloying (MA) combined with consolidation methods are an important techniques for preparation of titanium aluminides compacts with an ultrafine grain structure [5-8]. Therefore, γ titanium aluminides produced by MA are expected to exhibit improved workability at room temperature. Several techniques of the consolidation of milled powders were applied for obtain a high degree of densification retaining nanocrystalline microstructure [9]. The major problem is the choice of

Corresponding author: J. Dutkiewicz; e-mail: nmdutkie@imim-pan.krakow.pl

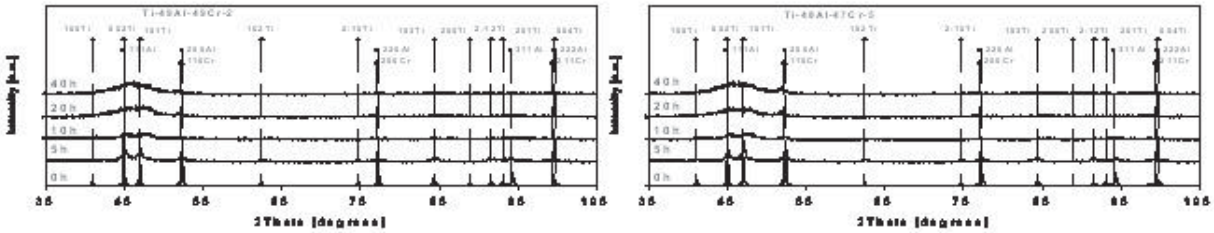

**Fig. 1.** Set of X-ray diffraction pattern of Ti-49Al-49Cr-2 (left) and Ti-48Al-47Cr-5 (right) alloys after different milling times.

process parameters and equipment for high temperature consolidation under relatively high pressure. The aim of this work was to determine the effect of consolidation process on the structure and mechanical properties of γ-TiAl-Cr intermetallics subjected to ball milling process.

## 2. EXPERIMENTAL PROCEDURE

Powders of titanium (110 μm size and of purity > 99.5%), aluminum (150 μm size and of purity 99.5%) and chromium (10 μm size and of purity > 99.8%) were used as starting materials. The powders were initially blended to the desired compositions of Ti-49Al-49Cr-2 and Ti-48Al-47Cr-5 (numbers indicate at.%) under argon atmosphere in a glove-box equipment and subjected to milling up to 40 hrs. A detailed description of the starting material and results of MA process are given in Ref. [10]. Subsequent hot pressing (HP) and pulsed plasma sintering (PPS) of the MA powder were applied. The uni-axial HP was performed at 1273K under 35 MPa for 1 hour in argon flow atmosphere whereas PPS conditions were as follow: impulse duration – 50 μs, maximum discharge voltage – 8 kV, initial pressure – 60 MPa, sintering pressure – $2 \cdot 10^{-2}$ Pa and sintering time 3 min. The powders used for PPS were initially degassed and reaction of sintering was initiated with 5 kV discharge voltage for 3 min. The structure changes during milling as well as of consolidated samples were studied a Philips PW 1830 diffractometer using Mo $K_\alpha$ radiation and transmission electron microscopy (TEM) in a Philips CM 20 equipped with a Phoenix energy-dispersive X-ray analysis system. Thin foils of pressed samples were prepared by dimpling and ion milling using Gatan equipment. The Vickers microhardness tests under the load of 10 N were performed for consolidated samples. The mechanical properties of HP and PPS samples in temperatures range from 293K up to 1073K was determined by compression testes with strain rate of $4 \cdot 10^{-4} s^{-1}$.

## 3. RESULTS AND DISCUSSION

As the staring material for consolidation a ball milled powders were used. The 40 hours long, high energy milling of Ti-49Al-49Cr-2 and Ti-48Al-47Cr-5 resulted in formation the mixture of almost fully amorphous phase and nanocrystalline Cr(Ti, Al) solid solutions. Fig. 1 presents X-Ray diffraction patterns of Ti-48,Al-47,Cr-5 alloy after different milling time. It shows a gradual disappearance of peaks from initial elements and formation of the amorphous broad peak in both alloys investigated. Only a low intensity peaks from bcc chromium can be seen after the longest milling time.

The final powder particles size was about 20 μm after 40 hours of milling. Analytical transmission electron microscopy allowed to identify the amorphous band structure as a matrix and small particles of Cr(Ti, Al) solid solution of size ranged from 10 to 25 nm [10].

The different consolidation methods of milled powders resulted in a different degree of densification but almost the same micro-hardness of

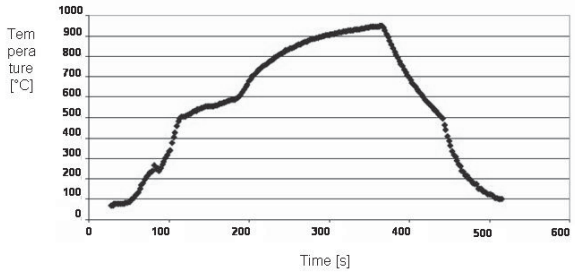


**Fig. 2.** Temperature vs. time characteristic during pulsed plasma sintering process.

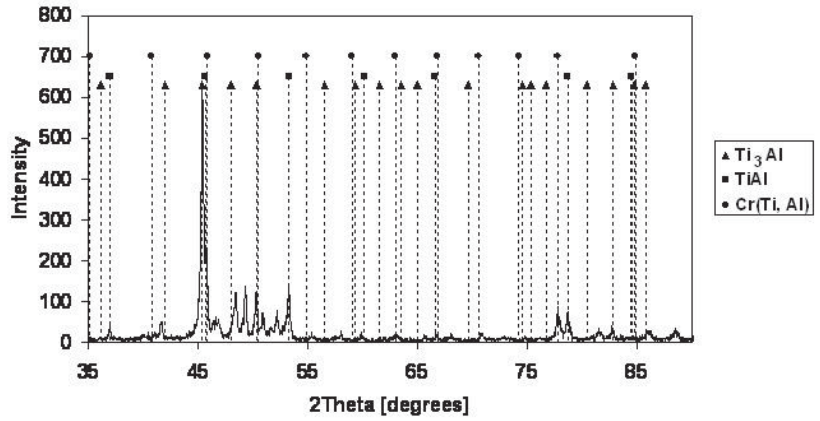


**Fig. 3.** X-Ray diffraction pattern of hot pressed Ti-48Al-47Cr-5 sample.

samples. The mean micro-hardness of samples consolidated by both methods was about 850 $HV_1$, whereas the density was about 95 and 98% of the theoretical one for HP and PPS samples, respectively. The mean transient temperature of PPS process was about 1223K (Fig. 2) and it was slightly lower then that of the HP (1273K) process. Therefore the differences in density can be attributed to almost twice higher pressure applied during PPS process. Additionally, the duration time of PPS process (Fig. 2) was very short in comparison to HP process what influenced on the final grain size, structure and mechanical properties of consolidated samples.

Fig. 3 shows X-Ray diffraction pattern of HP Ti-48Al-47Cr-5 sample. The three phase structure is mainly composed of $\gamma$-TiAl phase with a minor content of $\alpha_2$-$Ti_3Al$ and Cr(Ti, Al) phases. The unmarked peaks in the X-ray diffraction curve may be most probably attributed to the aluminium oxide. The analytical transmission microscopy investigations confirmed above observations. Fig. 4 presents elemental mapping and corresponding Bright Field (BF) micrograph of HP Ti-48Al-47Cr-5 sample. In addi-

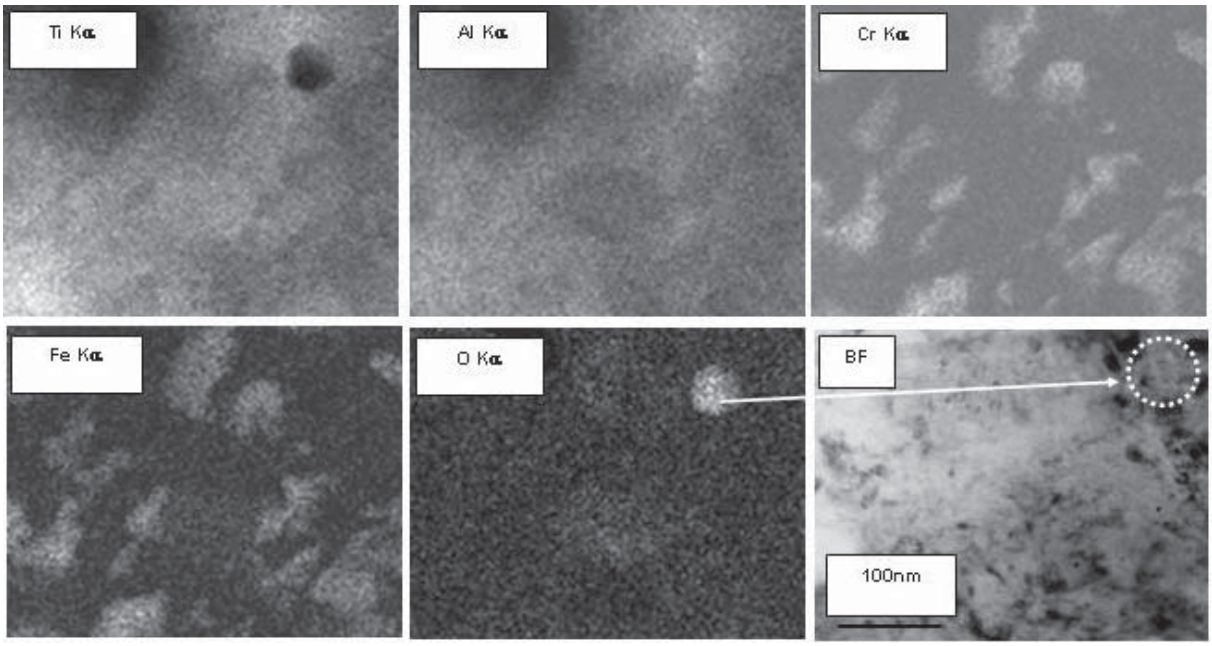


**Fig. 4.** Elemental mapping and corresponding bright field (BF) TEM micrograph of hot pressed Ti-48Al-47Cr-5 sample.

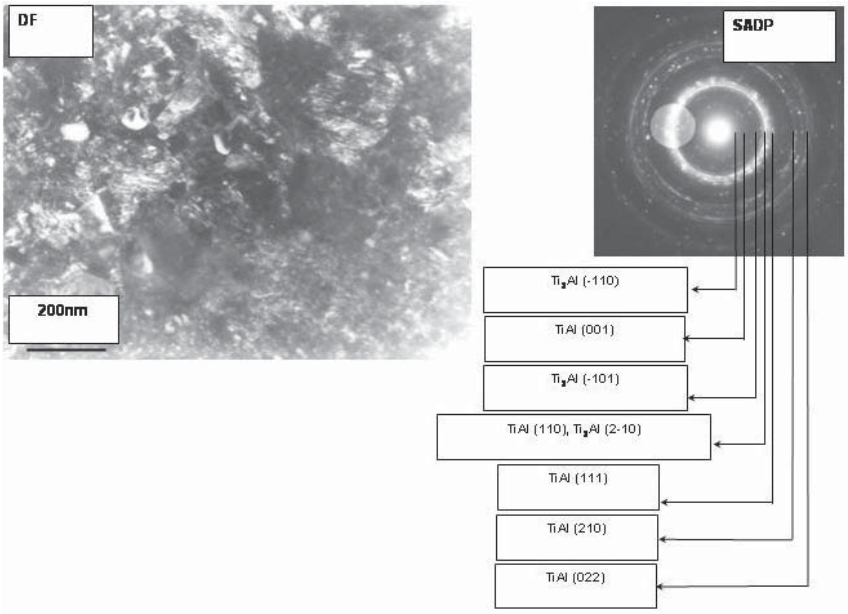


**Fig. 5.** Dark field TEM micrograph and corresponding SADP of hot pressed Ti-48Al-47Cr-5 sample.

tion to the matrix composed of homogenously distributed Ti and Al one can observe also the Cr rich and aluminium oxide particles. The size of aluminium oxides can be estimate as about of 50 nm. However, taking into account the results of the selected area electron diffraction (Fig. 5), the diffracted rings can be attributed mainly to $\gamma$-TiAl and $\alpha_2$-$Ti_3Al$ phases, what indicates that Cr is in solution in these phases and the amount of either Cr(Ti, Al) or aluminum oxides is very small. The Dark Field (DF) micrograph shows that the mean grain size in this sample is scattered from 50 up to 250 nm and for some larger grains the specific moire contrast was also observed. In the BF image presented in Fig. 6 one can see that the mean grain size of $\gamma$-TiAl phase is about 200 nm and they are mostly of equiaxed shape. The microstructure of PPS samples was more refined and characterized by large amount of twinned $\gamma$-TiAl grains; however, the same crystallographic structures like in HP samples were identified (Fig. 7).

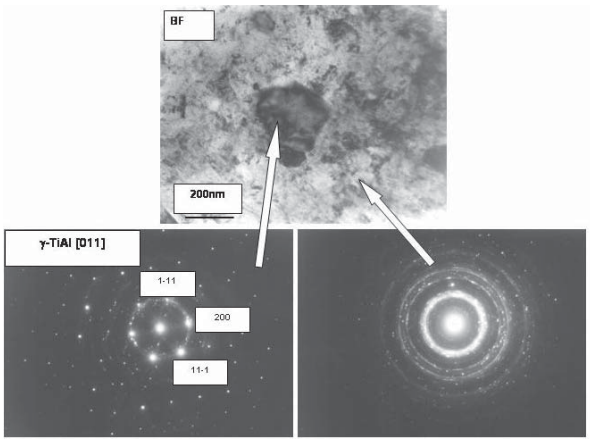


**Fig. 6.** Bright field TEM micrograph and corresponding SADP of hot pressed Ti-48Al-47Cr-5 sample.

In Fig. 8 one can see the straight type of boundaries in two neighboring grains. The left one corresponds to the twinned $\gamma$-TiAl of $[10\bar{1}]$ zone axis orientation with (111) twin plane. Its size is about 150 nm. The right grain shows lamellar $\alpha_2/\gamma$ grain boundaries of thickness of about 10 nm and zone axis orientations $[11\bar{2}0]$ $\alpha_2$-$Ti_3Al$ and $[1\bar{1}1]$ $\gamma$-TiAl. The interface boundaries are parallel to (0001) $\alpha_2$-$Ti_3Al$ and (111) $\gamma$-TiAl planes. Figs. 9 and 10 show compression tests curves of alloys hot pressed (Fig. 9) and pulse plasma sintered (Fig. 10). One can see that at room temperature compression strength of HP sample is only about 450 MPa, while that of PPS sample close to 1600 MPa. This difference results most probably from higher porosity of HP samples where crack initiation can be expected. Similar strength like that for PPS samples was observed by Ohering *et al.* [7] who used hot isostatic pressing (HIP) method for consolidation of ball milled Ti-Al-Cr powders. The PPS sample shows also higher

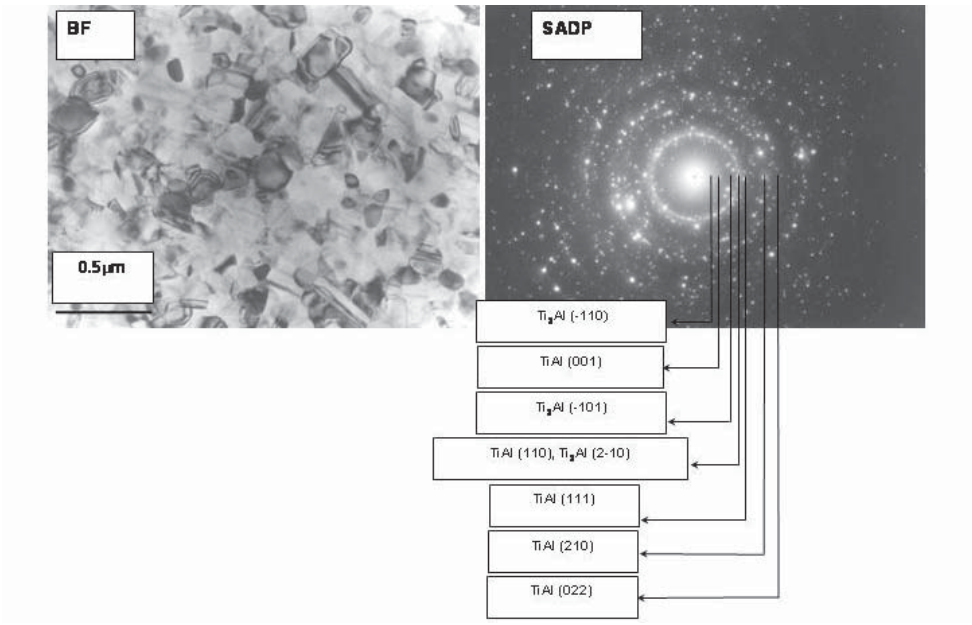


**Fig. 7.** Bright field TEM micrograph and corresponding SADP of pulsed plasma sintered Ti-48Al-47Cr-5 sample.

ductility at RT. The compression tests performed at 1273K indicate that for both samples the maximum strength reaches about 80 MPa, however the elongation of PPS samples is almost twice higher than that of HP one.

## 4. CONCLUSIONS

1. Addition of chromium up to 5 at.% into γ-TiAl alloy decreases its ability to amorphisation by ball milling, due to the a solid solubility of chromium in γ-TiAl. The nanocrystals of Cr(Ti, Al)

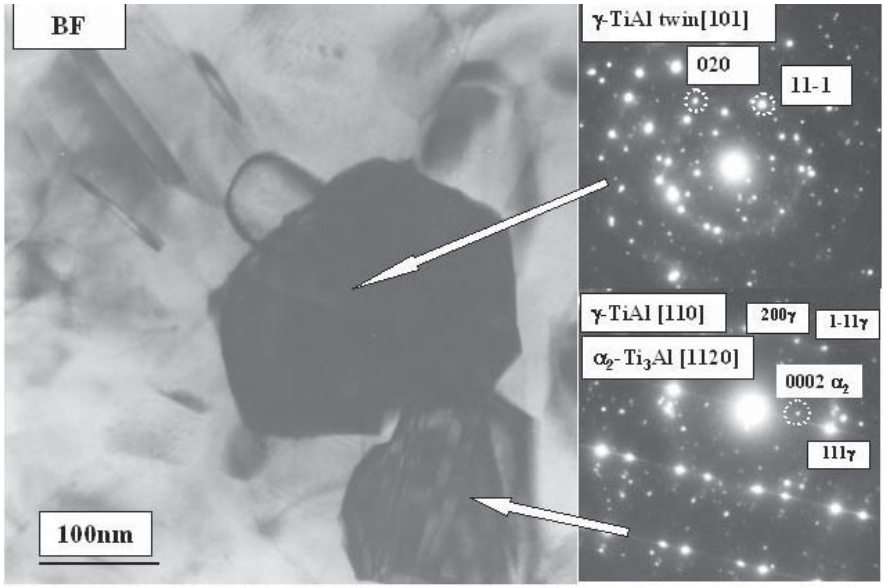


**Fig. 8.** Bright field TEM micrograph taken at higher magnification and corresponding SADP of pulsed plasma sintered Ti-48Al-47Cr-5 sample.

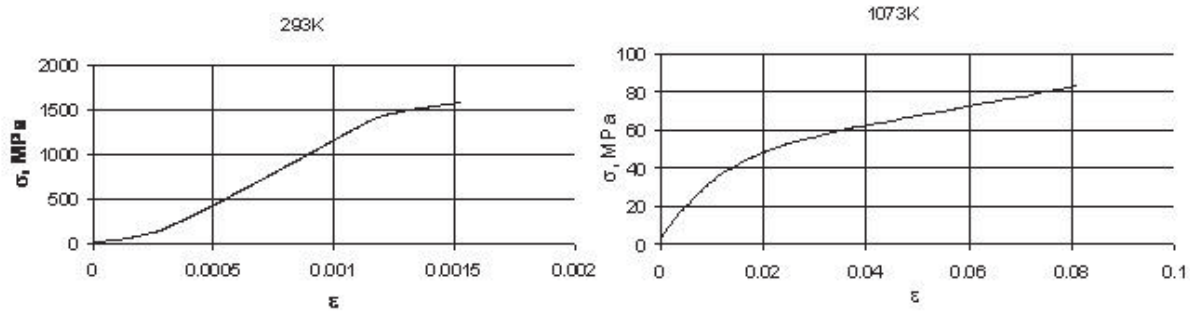


**Fig. 9.** Compression test curves at 293K and 1073K of hot pressed Ti-48Al-47Cr-5 samples.

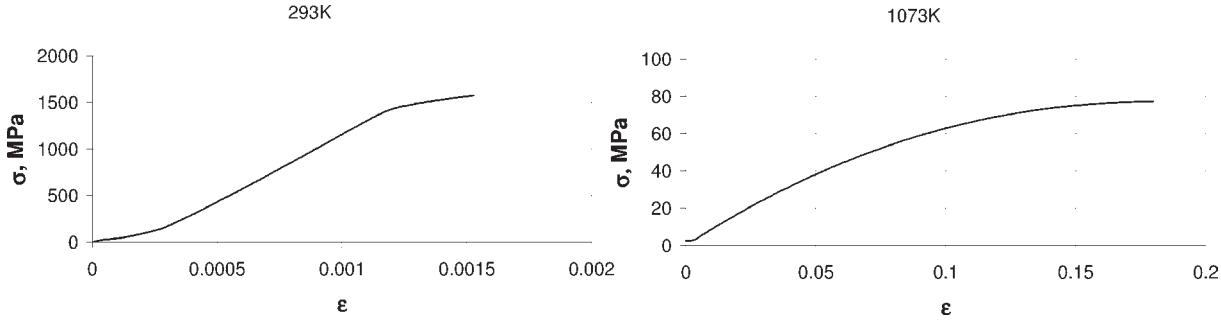


**Fig. 10.** Compression test curves at 293K and 1073K of pulsed plasma sintered Ti-48Al-47Cr-5 samples.

solid solution within the amorphous matrix were identified using transmission electron microscopy after ball milling process.

2. The ball milled powder, hot pressed at 1073K and pressure of 35 MPa, possess crystal size of about 250 nm and micro-hardness 870 HV. It consists of $\gamma$-TiAl, $\alpha$-$Ti_3Al$, and Cr(Ti, Al) solid solution grains. Its porosity is about 4% and show very low ductility during compression test at room temperature. The ductility increases at higher temperatures.
3. The ball milled powder sintered by pulse plasma method shows similar size of crystals, however with better defined grain boundaries and higher volume fraction of the $\gamma/\alpha_2$ lamellar structure. It shows similar hardness, but higher density and higher strength and elongation during compression tests at room temperature as compared to HP samples. The compression strength at elevated temperatures is similar to that of HP samples. The lower sintering temperature, the higher hardness is obtained.

## ACKNOWLEDGEMENT

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