Critical Revision of Experimental Data on Simultaneous Increase of Strength and Ductility in SPD pure metals

Alexander P. Zhilyaev [1,2 *], Yi Huang [3], Jose Maria Cabrera [4] and Terence G. Langdon [3]
[1]Fundació CTM Centre Tecnològic, Manresa, Spain
[2]Institute for Metals Superplasticity Problems, Ufa, Russia
[3]University of Southampton, Southampton, UK
[4]Universitat Politècnica de Catalunya, Barcelona, Spain
*E-mail: alex.zhilyaev@hotmail.com

Abstract
Already for fifteen years many researchers have been trying to discover metallic materials with unusual combinations of strength and ductility: with high strength and enhanced ductility. This report is an attempt to clarify the strength-ductility paradox by repetition of experimental work performed on HPT titanium of commercial purity. Experimental results for HPT bulk and powder titanium have demonstrated that there is nothing unexpected or unusual in mechanical behavior of CP titanium tested in HPT condition or after short annealing at 250 and 300 °C for 10 minutes. Moreover, high strength in HPT bulk titanium has a rational explanation due to the formation of hard omega phase during HPT processing at room temperature which transforms back to nanograined alpha phase domains during short annealing at elevated temperatures. HPT consolidation of titanium powder leads to creation of brittle specimens showing high strength but almost zero plasticity.

1. Introduction

This intriguing story begun almost 15 years ago, when Valiev with co-authors published a report [1] which has become highly cited to date. In this report the term "strength – ductility paradox" was firstly introduced for describing enhanced plasticity in HPT titanium processed for 5 whole revolutions compared to that processed for one revolution [2]. Note that specimens have the same strength (~ 800 MPa) and they were tested at 250 °C. The aim of this report can be articulated as follows: we intend to examine the "strength-ductility paradox" for commercially pure titanium by consolidating titanium powder at room temperature by HPT in order to obtain Ti-TiO composite and performing microstructural analysis and mechanical testing. Experimental results will give us an answer on plausibility of the "strength-ductility" concept in UFG titanium. The results partially have been published elsewhere [3].

2. Experimental Procedures

Experiments were conducted on commercial purity (CP) titanium powder having a mesh size of ~150 μm obtained from Goodfellow Ltd., Cambridge, UK. The powder was pre-compacted and further consolidated by HPT under an applied pressure, P, of 6.0 GPa, using a rotation speed of 1 rpm and torsional straining through numbers of revolutions, N, of 5 turns. Microhardness, XRD and TEM analysis as well as tensile testing were performed on the powder consolidated and bulk Ti disks. The mechanical properties were examined at room temperature and at elevated temperatures of 250 and 300°C. All elongations were carefully calculated by measuring the gauge lengths before and after tensile testing using an optical microscope.

3. Results and Discussion

The microhardness distribution along the diameter of the HPT-processed Ti powder compressed to a disk is given in Fig. 1. It is seen that after pure compression without torsional straining (P = 6.0 GPa, N=0 and loading time of 1 min) and annealing at 700°C for 40 min the microhardness distribution is highly inhomogeneous along the disk diameter. This inhomogeneity is so large that there is a difference in Hv of up to more than 100%. By contrast, after HPT processing for 5 whole revolutions the microhardness is essentially fully homogeneous and equal to Hv ~300. This is consistent with earlier results [4] where the value of the microhardness for consolidated BM titanium powder was Hv ~ 350.

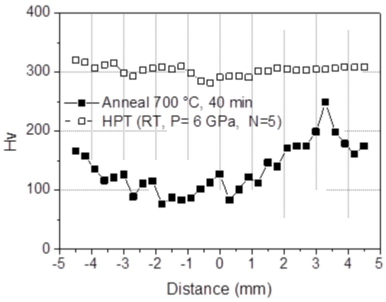

Fig.1 Microhardness, Hv across the disk diameter of Ti powder-consolidated disk annealed and HPT processed.

The XRD results on the evolution of the crystallite size and microstrain of the samples of HPT-consolidated Ti powder are shown in Fig.2: evidently the microstrain has a reciprocal trend, increasing to a maximum for HPT-consolidated disks of titanium and decreasing during annealing. It is worth noting that during the short annealing at 250°C the microstrain decreases more than 2 times whereas the crystallite size increases only by ~20% from 36.9 nm to 42.4 nm. During annealing at 300°C the microstrain decreases slightly but there is a continuously increasing crystallite size. Apparently at the lower temperature (250°C) mostly a relaxation of the microstrain takes place whereas at higher temperatures (300°C) some growing of the crystallites (subgrains) can occur.

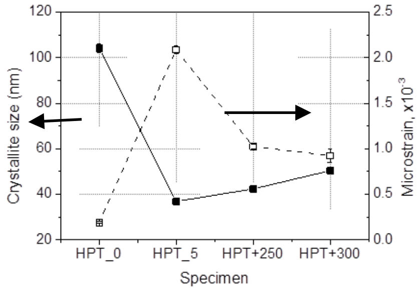

Fig.2 Crystallite size and microstrain as a function of the sample conditions: HPT_0 corresponds to the compression at P=6 GPa, N=0, loading for 1 min; HPT_5 corresponds to HPT straining of Ti powder at P=6 GPa, N=5; HPT+250 and HPT+300 are HPT specimens with annealing for 10 minutes at 250 and 300 °C, respectively.

The fine microstructure of the Ti-consolidated powder after HPT processing and short term annealing is shown in Fig. 3, where as an example the HPT-consolidated titanium annealed at 300°C for 10 min is shown.

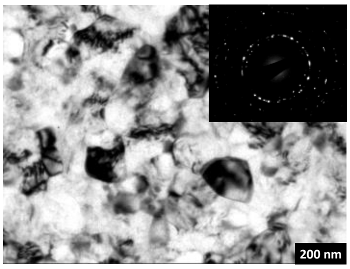

Fig.3 Bright field TEM of Ti powder consolidated by HPT (P=6 GPa and N=5 and annealed at 300 °C for 10 minutes). Inset depicts SAED.

The mean grain size, *d*m, was calculated as *d*m ≈ 202.5 ± 71.9 nm for the HPT-consolidated disk of powder titanium, 213.9 ± 63.6 nm for the sample annealed at 250°C and 237.0 ± 69.1 nm for the disk annealed at 300°C. Due to the relatively small statistics, these results have a high dispersion. Nevertheless, a trend of increasing grain size is evident. These values correspond to the trend observed from the XRD results, with minimal (sub)-grain growth at the lower annealing temperature (250°C) and noticeable growth at the higher annealing temperature (300°C).

Representative plots of engineering stress against engineering strain are shown in Fig. 4 for the HPT-consolidated titanium powder. It is intriguing to note that all specimens except for the sample annealed at 300°C for 10 min show zero ductility. However, it is apparent from Fig. 4 that the specimen annealed at 300 °C (10 min) showed enhanced plasticity of ~2.2%. In practice, this is lower than the plasticity of ~8% reported for the consolidated BM powder subjected to 10 whole revolutions [4]. It is worth noting that apparently during HPT straining to 10 revolutions the titanium underwent a reverse omega to alpha phase transformation which can increase the final ductility.

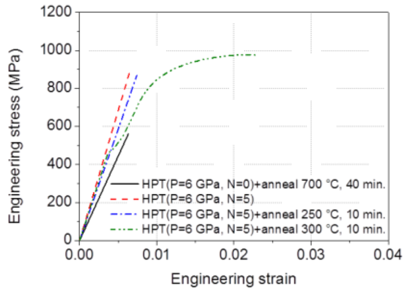

Fig.4 Engineering stress/strain plots for powder Ti subjected to HPT (RT, P=6 GPa, N=5) and additional annealing for 10 min at T=250 and T=300 °C. Black dot line is tensile plot for the samples annealed at 700 °C for 40 min. All annealing was performed in vacuum (10^{-3} torr.). Strain rate was 10^{-3} s^{-1}.

4. Conclusions

The consolidated HPT Ti samples had an ultrafine grain size, a nanometer crystallite size, a very high level internal stress and a correspondingly high dislocation density. As a result, the behavior was generally brittle in tensile testing. Short annealing for 10 minutes at 250°C led to a relaxation of the internal stress and no significant (sub)-grain growth. Annealing at the higher temperature of 300°C led to a significantly increased ductility up to ~2.2 %.

Acknowledgments

APZ acknowledges a TECNIOSPRING grant financed by Generalitat de Catalunya and co-funded by the 7th Framework program of the EU

References

[1] R.Z. Valiev, I.V. Alexandrov, Y.T. Zhu and T.C. Lowe: *J. Mater. Res.* **17**(1) (2002) 5-8.

[2] R.Z. Valiev, A.V. Sergueeva, A.K. Mukherjee: *Scripta Mater.* **49** (2003) 669-674.

[3] A.P. Zhilyaev, G. Ringot, Y. Huang, J.M. Cabrera, T.G. Langdon: *Mater. Sci. Eng. A.* **688** (2017) 498-504.

[4] C.T. Wang, A.G. Fox, T.G. Langdon: *J. Mater. Sci.* **49** (2014) 6558-6564.