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Study on the Crystal Structure and Microstructure Evolution of Shock-processed Titanium Powder

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ABSTRACT

Titanium powder was rapidly solidified by using shock-wave consolidation technique. The critical parameters were controlled by instrumented detonics and pin-oscillography. The compacted specimens were investigated for crystal structure and microstructural strengthening by using standard diagnostic techniques. The density of the final product was found to be greater than 96% of the theoretical value. X-ray diffraction pattern reveals intact crystalline structure without the presence of any undesired phases. The particle size reduction indicated by XRD was supported by laser diffraction based particle size analyzer. Results from energy dispersive spectroscopy ruled out the possibility of any segregation within the compacts. Scanning electron microscopy showed crack-free, voids-free, melt-free, fracture-less compacts of titanium with a unidirectional dendrite orientation without any grain-growth.

1. Introduction

Titanium has a moderate strength to weight ratio, corrosion resistant properties and ability to withstand at elevated temperature without creeping and thereby making it a strong candidate to be serviceable in modest aerospace industry, lightweight engine shafts, motor parts and making critical components for strategic cryogenic applications in defence^[1,2]. Several powder consolidation processes can be adopted to obtain an extensive range of rapidly solidified (RS) compositions of titanium. Rapid solidification can affect the resulting crystal structure, microstructure, morphology, constitution and mechanical properties of the specimen. Conventional compaction methods applied in powder metallurgy exert high pressure and temperature

on the specimen for long duration^[3]. This prolonged temperature exposure may result into grain-growth, melting of the compacted specimen and deteriorate the fine-grained structure that can reduce the mechanical properties.

As such the consolidation temperature must act relatively for lesser time. Therefore, innovative technique like shock wave compaction (SWC) has been developed. SWC is an emerging technology wherein fine powders are dynamically compressed to produce monoliths of desired dimensions. Shock wave being transient in nature requires no additional binders. With a working time of few micro-second, it offers opportunity to consolidate the fine powder particles without any grain-growth. The dynamic high pressure of shock wave causes pore-elim-

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ination whereas the transient high temperature leads to surface heating, resulting into interfacial locking of the particles while keeping the particle interior intact from high temperature exposure^[4]. Figure 1 represents different processes ongoing during the SWC of powders. The present study aims at producing compacted specimen of pure titanium powder under shock-wave loading while retaining its crystal structure without formation of undesired phases and obtaining a better microstructure without grain-growth, without melting or without segregation. The objective is to correlate theoretical considerations with experimental analysis as well.

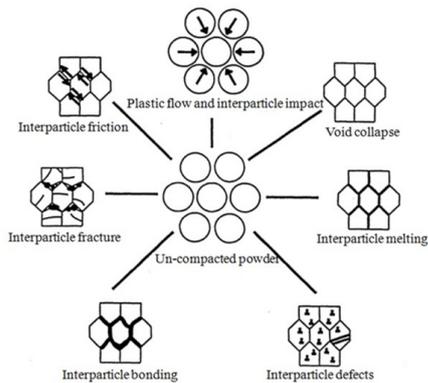


Figure 1. Various modes of energy dissipation in SWC of powders

2. Experimental Work

The experimental work comprises two parts: one involves the compaction phenomenon and the other concentrates on the analysis of the specimen using standard diagnostic techniques. Figure 2 represents a block diagram of the SWC system. It consists of a cylindrical ampoule with a conical cap at top and a plane cap in the bottom of the cylinder. Titanium powder is filled in this cylindrical ampoule with special care that air may not remain entrapped between the fine-grained particles. This cylindrical geometry is now put inside a perplex pipe filled gently with an explosive material. A detonator covers the top of the specimen-loaded arrangement. A pin oscillographic technique including a pulse generator (pulser) and a Digital Storage Oscilloscope (DSO) was used to measure velocity of detonation. The choice of explosive is very critical aspect for SWC as it involves detonation wave, shock wave and expanding gases that may directly affect the corresponding microstructure of the specimen under consideration. An explosive called trimonite with a moderate velocity of detonation is used for this specific purpose. The whole configuration placed in an earth pit is detonated with the help of exploder dynamo condenser (EDC). The detonation

wave on combination with explosive produces a spherical shock-front that swiftly travels down to the compaction system. The spherical shock-front becomes plane wave-front after passing through the conical top. This planer shock-front travels gradually down to the ampule and is responsible for the compaction of the powder^[5].

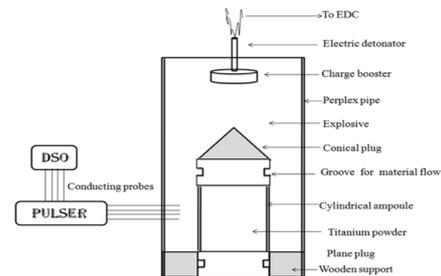


Figure 2. Compaction geometry for SWC of titanium powder

X-ray diffraction (XRD) technique was employed to determine the crystal structure, phase and FWHM values. The device used was X'PERT-PRO X-ray detector with a fixed divergence slit operating at 45kV and 40mA, with a step-time of 1.0s and step-angle 0.02° using Cu K α lines having wavelength of the order of 1.54Å. The goniometer was varied through the angle (2 θ) from 10° to 80°. The variation in particle size of the shock-processed specimen was examined with the help of laser diffraction based particle size analyzer (Malvern mastersizer, S-2000) having lens range 300RF and a beam length of 1.9mm working at wavelength of 633nm. The chemical homogeneity and segregation within the compacted specimen were examined by using energy dispersive spectroscopy (EDS, Genesis APEX4) with a single shot multibox detector, SSD Apollo10, at energy ranging from 0-30 keV. To determine the surface morphology, core microstructure, melting, fractures and cracking of the shock-processed specimen, scanning electron microscopy, (SEM) FEI, Quanta 250, D-9393 operating at 20 kV was used.

3. Results and Discussion

3.1 Determination of crystal structure and phases

Figure 3 represents graphic workspace fit X-ray diffraction pattern of un-processed and shock-processed titanium powder. XRD study reveals that the shock-processed titanium is much similar to the un-processed specimen. The diffraction peaks (001), (100), (101), (110), (102) and (111) confirm hcp crystal structure of titanium. After being indexed, the peak position of shock-processed specimen found to possess same crystalline phase as that of un-processed specimen. No extra peak was observed in the compacted specimen that rules out the possibility

of impurity and presence other phases. It can also be seen in the X-ray pattern that FWHM values of the shock-processed specimen have been increased significantly (from 0.4793 to 0.9507⁰2θ). One can conclude from the well-known Scherrer's formula that grain size has been reduced in the compacted specimen. This broadening in the diffraction peaks attributes to a decrease in crystallite size under intense shock-loading. The slight shift of the diffraction peaks towards the higher angle also point towards the decrease in lattice parameters (Bragg's law). This shift in peak position may be attributed to a few crystalline defects owing to the melting of particle interfaces [6,7]. More intense peaks in the diffraction pattern of shock-processed specimen indicate the more density of the particles at that particular position or plane. A careful observation of the X-ray depicts that there is small disturbance or we may say small peaks around the 52⁰ and 64⁰(2θ) in the shock-processed specimen whereas no such disturbance/peak is observed in the pure titanium powder [8]. These peaks point towards the oxide formation in the compacted specimen. The justification stems from the fact that air may remain entrapped during the tapping of the powder in the cylindrical ampoule resulting into oxide formation. Oxide formations in the compacts are responsible for lower density of the final product [9,10].

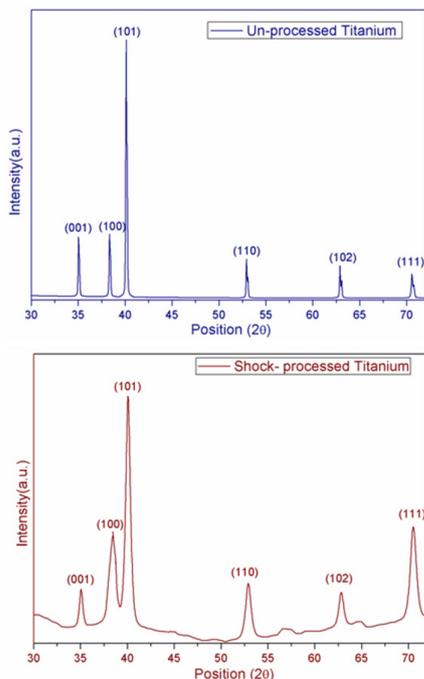


Figure 3. Graphic workspace fit XRD pattern for un-processed and shock- processed titanium

3.2 Determination of Particle Sizes

To support the reduction in particle size in the compacted

specimen revealed by the X-ray diffraction method, we used laser diffraction method. It applies small angle laser light scattering principle to measure particle size distributions of powder. Figure 4 shows Gaussian fit particle size distribution (logarithmically normal) for un-processed and shock-processed titanium. Table 1 represents the data recovered from laser diffraction method. D(v,0.9) reflects 90% of the sample is under this size. Similarly, D(v,0.1) means 10% of the sample is below this size and likewise D(v,0.5) denotes 50% of the particles lie below this size range. The volume moment mean diameter D(4,3) was calculated by using the relation: $\bar{D}_{(4,3)} = \frac{\sum V_i D_i^4}{\sum V_i D_i^3}$. Where, V_i is the volume of particles and A_i is actual surface area of the particles called de-Broncker mean diameter [11].

The density of the shock-processed specimen found nearly the theoretical value (4.506g/cm³). That means SWC technique is quite helpful to achieve a density more than 96% of the actual value. For un-processed powder specimen, average moment mean diameter was found to be 32.56μm which had been reduced down to 24μm.

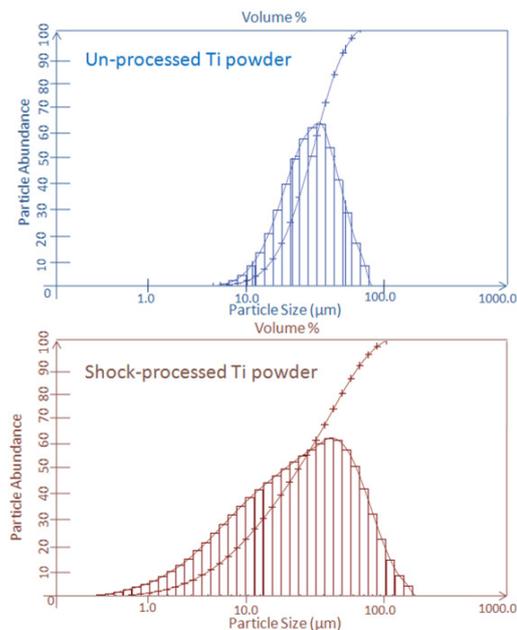


Figure 4. laser diffraction based particle size distribution for un-processed and shock-processed titanium

Table 1. Data recovered from laser diffraction on the pure and shock-processed titanium

Parameters	Un-processed Ti	Shock-Processed Ti
Specific Surface Area	0.1395m ² /g	0.0586 m ² /g
D [v,0.9]	48.06 μm	40.25 μm
D [v,0.1]	10.80 μm	12.40 μm
D [v,0.5]	27.65 μm	24.35 μm
D [4, 3]	32.56 μm	24.03 μm

3.3 Determination of Elemental Composition and Segregation

There may arise some problems with respect to the segregation of impurity elements and coarse inter-metallic compounds due to the formation of new phases in SWC process. Better microstructure controls the segregation phenomenon and dispersed position of non-metallic inclusions. Therefore, energy dispersive spectroscopy is used to explore compositional changes and segregation within the compacted specimen.

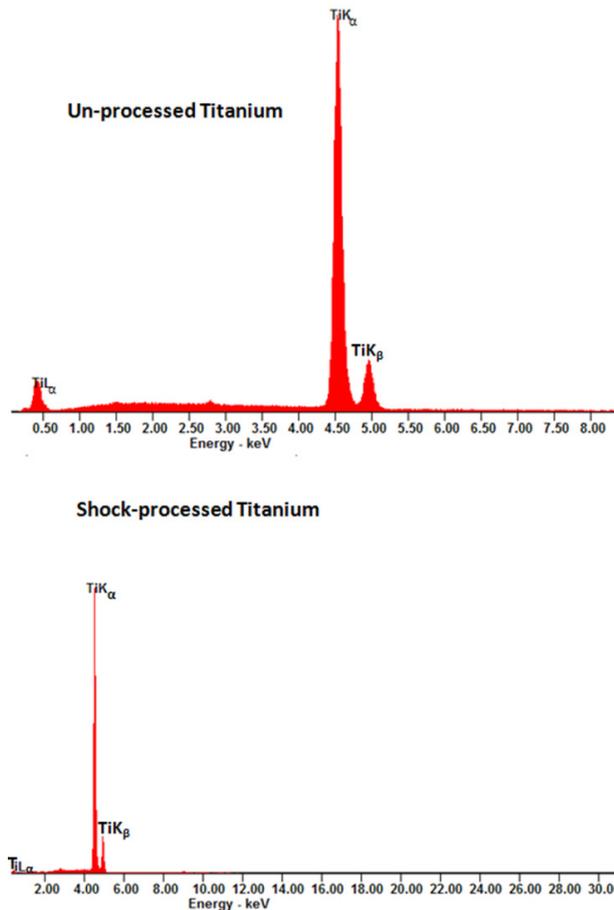


Figure 5. EDX data indicating no segregation within the compacted specimen

Figure 5 shows the energy dispersive spectra of pure titanium (99.96% purity) and shock-processed Titanium. Quantitative EDX data suggested no segregation within the compacts after the shock-loading. In fact, the sample under investigation was put into large energy range upto 30keV but only three peaks TiK_{α} , TiK_{β} and TiL_{α} were observed with the same peak positions. This means that the sample possess chemical homogeneity. Since, the rapid solidification occurs at quench rates of the order of 10^5 K/s for particles of $100\mu\text{m}$ or less^[12]. This high-quenching during rapid solidification minimize chemical segregation

and formation of massive phases and hence ensures a homogeneous fine-grained structure.

3.4 Determination of Morphology and Microstructure Evolution

Figure 6(a) is an SEM picture of pure titanium powder. The particles are primarily of various sizes and morphologies. The particles are rough needlelike structure. Some particles appeared to be crystalline while others appeared amorphous. Even after size classification, large variations in particle morphology and structure were observed. The particle-size of the titanium powder calculated from SEM image found to be $\sim 30\mu\text{m}$ that supports the earlier observation. Figure 6(b) concentrates on a single particle at a higher magnification (4000 \times). The morphology of the particle consists of uneven smooth surface. It reflects distribution fine dendritic structure on the surface of particle in different directions. Such irregularities are favorable for SWC as it may produce better interlocking between particles and lead to higher density of the final product.

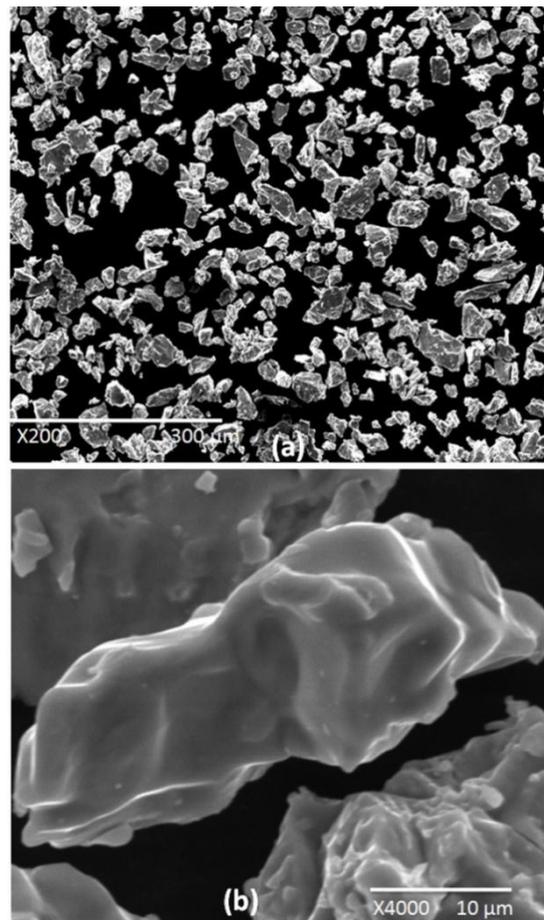


Figure 6 (a). SEM image of pure titanium powder particles. (b) magnified image of a single particle showing its surface morphology

Working with shock-loading of metal powders faces a major problem with melting of the final product. Therefore, micrograph images were taken from the inner portion of the shock-processed specimen at different magnifications to realize these issues. Figure 7(a) and (b) demonstrate core fracture morphology of the shock-processed titanium with unidirectional heat-flow. It can be seen from higher magnified image that dendrites are oriented in the direction of heat-flow within the compacts. Such structure supposed to possess good mechanical properties. Distinctive grain boundary can be seen at higher magnification (1200×). Two sub-structurally different regions can also be observed in figure 7(c) and (d), respectively. Regions marked with “A” showing only little cracking of the compacts. Cracking may be due to uneven load under deliberate breaking of the compacts to observe these regions within the core of compacted titanium. Cracking within the compacted specimen is attributed to tensile/compressive stresses, and shear instability or solidification shrinkage.

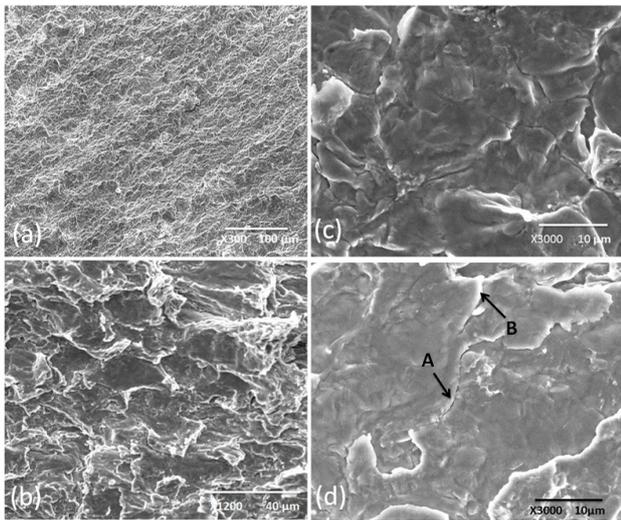


Figure 7 (a). heat-flow throughout the surface of compacted specimen. (b) higher magnified image showing dendrite orientation within the inner portions of compacts. (c) fractograph showing the well-bonded structure in the core of compacted specimen surface (d) microstructure across the grain boundaries

No micro-void was observed in the whole compacted specimen. The regions marked with ‘B’ correspond to melting across particle interfacial. These melting layers are responsible for the bonding between the particles under the intense deposition of shock-energy. Because shock pulses being transient in nature apply only for few micro-seconds, the melted layers re-solidify instantly and the grains do not respond against their growth during SWC process. Thus, the apparent white regions across the

grain boundaries are produced due to rapid- solidification shrinkage^[13]. The grains of the shock-processed specimen seem to be of smaller size as that of un-processed specimen shown by figure 7(d). It is clear that shock wave loading considerably deformed the particle shapes as well. These irregularities increase the contact area of the specimen and hence the strength considerably. Such sub-structurally unique situations under controlled parameters lead to better microstructures that provide better end products^[14].

4. Conclusions

SWC technique helped in forming the uniform melt-free, crack-free, voids-free compacts of titanium powder. The crystalline structure of the compacted specimen remained intact. No impurity or phase of any other kind was detected. There is no segregation within the compacts. A remarkable particle size reduction was observed that rules out the possibility of grain-growth during compaction process. The density of the final product was found to be more than 96% of the theoretical value. Shock-wave consolidation being transient in nature, owing to its short processing time, controlled parameters and high quench-rates proven to be a very helpful technique for obtaining a stable structural and microstructural products.

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