Sintering Behavior of Mechanically Alloyed Titanium – Titanium Nitride Nanocomposite Powders

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Abstract

The sintering behavior of titanium-titanium nitride nanocomposite powders has been studied by dilatometry. Titanium – titanium nitride nanocomposite powders were produced by the reactive milling of micron sized titanium powder (12μm) in nitrogen atmosphere. The Ti-TiN nanocomposite powders milled for various durations along with the initial micron sized Ti powders were then sintered in the temperature range of 450-1000°C by a constant rate of heating (10°C/ min). The linear shrinkage, shrinkage rate, activation energy for sintering and microstructure has been studied and discussed as a function of milling time.

Keywords : titanium, sintering, nanocomposite, mechanical alloying

1. Introduction

Nanocomposites in general are those materials in which either the reinforcement or the matrix or both are in the nanometric range (<100 nm). The development of nanocomposites has emerged as an important step in creating a new generation of materials. The reduction in the particle / grain size of these materials results in superior properties when compared with their coarse particle / grain size counterparts: higher strength / hardness, enhanced diffusivity, improved ductility / toughness, etc [1-3].

In recent years there has been considerable interest in the development of nanocomposites by the consolidation of nanocomposite powders. The most challenging problems in nanocomposite powder consolidation is grain coarsening which results in the loss of the desired nanostructure, high tendency for the nanocomposites powders to agglomerate, significant inter particle friction, and high reactivity and associated contamination [4]. The densification of nanocomposites is difficult due to the different sintering behavior of the parent matrix and the second phase. The densification rate of nanocomposites is not a linear function of the densification rates of the two components and each phase in the mixture has its own densification and coarsening rate. In crystalline matrices of nanocomposites, densification rates are lower than theoretical at all volume fractions [5,2].

The present investigation aims at studying the sintering behavior of titanium-titanium nanocomposite powders by dilatometry.

2. Experimental and Results

The Ti-TiN nanocomposite powders were synthesized on a laboratory scale by the reactive milling of micron sized Ti powder in nitrogen atmosphere. Micron sized Ti powder (99.7% purity) of average particle size of 12 μm particle size was milled in a high energy attrition mill for various durations of 15, 30, 45 and 60 h. The powders after degassing (to remove the process control agent) were characterized by XRD, SEM, TEM, DTA, XRF and ICP-AES. The particle size as measured by TEM was found to decrease exponentially with milling time. XRD revealed the formation of non- stiochiometric nitride phases at 15 h of milling. The titanium nitride content was found to increase with the milling time as revealed by XRF analysis. DTA revealed a drop in the α-β transition temperature with milling time while ICP-AES analysis showed an increase in the impurity content (Fe, Cr, Ni) with milling time.

The Ti-TiN nanocomposite powders as well as the initial micron sized Ti powder were sintered in a dilatometer (Shimadzu thermal analyzer) at a constant heating rate of 10°C/min at various temperatures in the range of 600-1000°C. From the dilatometric curves the shrinkage (y) vs temperature (T) curves were obtained. The shrinkage was in general found to decrease with the milling time. From the shrinkage the shrinkage rate (r) was determined. The shrinkage rate was an order higher in the milled powders as compared to the initial micron Ti powder. A plot of logarithm of shrinkage rate vs inverse absolute temperature i.e. plot of ln r vs 1/T, gave a curve whose slope is equal to the activation energy for sintering. Table 1. shows the obtained activation energy (Q) values. The activation energy values were in general found to decrease with the milling time in both the α-Ti and β-Ti ranges. These were also found to be lower in the β-Ti as compared to the α-Ti range.

Figure 1. shows the SEM photograph of the 60 h milled Ti-TiN nanocomposite compacts sintered at 1000°C. The densification level in the specimens was found to decrease with milling time while the presence of nitride particles was found to increase with milling time.

Table 1. Activation energy values for micron Ti (0 hr) and Ti-TiN nanocomposite powders milled for various durations.

System	Q (α-Ti) (kJ / mol)	Q (β-Ti) (kJ / mol)
0 hr	92 ± 6	70 ± 4
15 hr	104 ± 8	25 ± 2
30 hr	83 ± 6	30 ± 3
45 hr	64 ± 6	35 ± 2
60 hr	50 ± 6	23 ± 2

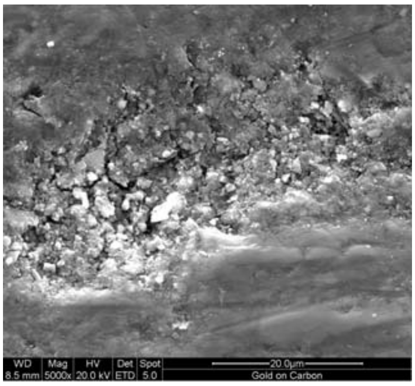

Fig. 1. SEM photograph of 60 h milled Ti-TiN nanocomposite powder sintered at 1000°C for 1 h.

3. Summary

The particle size, crystallite size, α-β transition temperature were found to decrease with the milling time while the nitride formation and impurity content was found to increase with the milling time. The shrinkage and the shrinkage rate was in general found to increase with the milling time. The activation energy for sintering was in general found to decrease with the milling time.

4. References

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