Preparation and Sintering Behavior of Fe Nanopowders Produced by Plasma Arc Discharge Process

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Abstract

The nano-sized Fe powders were prepared by plasma arc discharge process using pure Fe rod. The microstructure and the sintering behavior of the prepared nanopowders were evaluated. The prepared Fe nanopowders had nearly spherical shapes and consisted of metallic core and oxide shell structures. The higher volume shrinkage at low sintering temperature was observed due to the reduction of surface oxide. The nanopowders showed 6 times higher densification rate and more significant isotropic shrinkage behavior than those of micron sized Fe powders.

Keywords : Fe nanopowder, plasma arc discharge, sintering

1. Introduction

Interest in nanoscale materials has increased in recent years with the realization that unique properties may be obtained from an extrapolation of known grain size effects to nanograin sizes. The potential application of nanostructured powder materials as novel structural or functional engineering materials largely depends on the consolidation of nanopowders into nanoscale bulky materials. So the controlling of the powder characteristics could be a breakthrough for development of nanopowder consolidation.

The main research trend has been the realization of higher compaction density and lower temperature/shorter exposure time in sintering step to avoid the tremendous grain growth of nanopowders, using high pressure compaction and rapid/pressurized sintering techniques. However these techniques have a limitation for the near net shaping into the small sized, complex sintered components and mass production [1-4].

In this study, we have adopted the Plasma Arc Discharge (PAD) process for the fabrication of Fe nanopowders. The shrinkage behavior of Fe nanopowder was sytematically analyzed and compared with that of micron sized Fe powders.

2. Experimental and Results

Fe nanopowders were fabricated by plasma arc discharge process as described in our previous work [5]. The Fe metal vapors evaporated by plasma arc heat in the reaction chamber collided each other and condensed to form Fe clusters or nanopowders. For generating plasma arc, the applied current and operation pressure were 270A and 760 Torr, respectively. Pure Ar gas mixed with 10-50 vol. % H_2 was filled in the operation chamber. The Fe nanopowders were uniaxially pressed with 175-2,100 MPa in a cylindrical compaction die, to form disk shape specimens. The green densities of each compacted bodies were 43% and 62% theoretical density (T.D.), respectively. The shrinkage behaviors of compacted bodies were measured by using laser opto-dilatometry system developed by Lee and his coworkers [6]. The compacted bodies were heated up to 1000^{o}C in hydrogen atmosphere with various heating rates of 5, 10, 20 and 30^{o}C/min. The dimensional changes of diameter and height of each sample were measured and plotted against elevating temperatures. For comparison, micron sized Fe powders (5-10um, 99.9%) were compacted with 350 MPa (47%T.D. of green density) and sintered at 1200^{o}C for 2 hours in H_2 atmosphere.

The prepared Fe nanopowders are nearly spherical with a core-shell type structure and mean diameter about 70 nm. They were composed of pure Fe core and Fe oxide shell. The thickness of the shell is about 4-5 nm. The Fe nanopowders compacts were sintered under hydrogen atmosphere with different heating rates. The volume shrinkage of each sample was measured by a laser-opto dilatometry system, and the results are shown in Fig. 1 (a). As can be seen from this figure, the volume shrinkage rate can be divided into two regimes, i.e., higher rate at low temperature region and retarded rate at high temperature region. The higher volume shrinkage region occurred at the temperature range of 260-380^{o}C. For all the heating rates, the volume shrinkage rate is higher in low temperature region. This high volume shrinkage may be originated from the phase transformation such as oxide reduction, magnetic phase transformation, or accelerated nanopowder accommodation, because the densification cannot be active in the low temperature range of 260-380^{o}C. This was confirmed by thermogravimetry measurement (Fig. 1 (b)). The black line of weight loss and dotted line of the volume shrinkage line has been put together in one figure. As shown in the figure, the second weight loss region in the 300-420^{o}C agrees well with the higher volume shrinkage regime of Fig. 1 (a). This means that the volume shrinkage for oxide reduction is the dominant factor in higher volume shrinkage rate in relatively low temperature region. Based on the foregoing observations, we can conclude that the surface reduction of oxide layer of nanopowders plays an important role in the higher volume shrinkage in the early sintering of nanopowders, and is believed to be influential in the nanopowder sintering with the inhibition of grain growth.

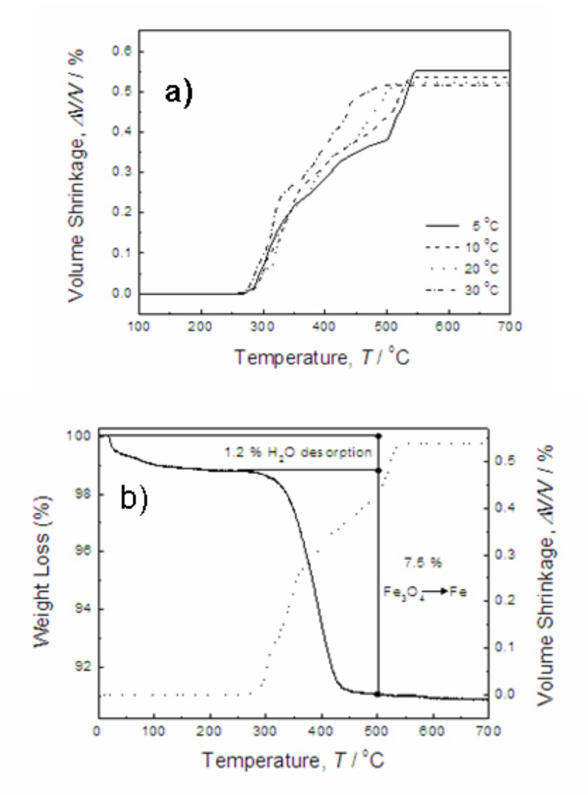

Fig. 1. Volume shrinkages of PAD nanopowder compacts with different heating rate in hydrogen atmosphere (a) and thermo-gravimetric curve (b) of nanopowder compact with volume shrinkage curve in hydrogen atmosphere with 10°C/min.

The shrinkage rate of nanopowders is 6 times higher than that of micron sized powders and the nanopowders show more significant isotropic shrinkage behavior. We believe that this is attributed to the non agglomeration and uniform size distribution of the PAD nanopowders. This means that the PAD nanopowders and subsequent presureless sintering process have a great potential for high strength sintered components which is suitable for fabrication of the near net shaped micro machinery with complex shapes.

3. Summary

The formation of Fe nanopowders by Plasma Arc discharge (PAD) process and pressureless sintering behavior of Fe nanopowder were investigated. The prepared Fe nanopowders consisted of metallic Fe core and oxide shell structures with nearly spherical shapes. In the nanopowder sintering, the reduction of surface oxide played an important role in the initial volume shrinkage and affected the total densification and grain growth process at a relatively low temperature. The Fe nanopowders showed 6 times higher densification rate and more significant isotropic shrinkage behavior than those of micron size Fe powder.

4. References

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