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Ultrasonic evaluation of the morphological characteristics of metallic powders in the context of mechanical alloying

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ABSTRACT

An ultrasonic method is proposed to characterize the morphological (geometrical) aspects of powders through the elastic modulus dependence of their packing on the factors of polydispersity, coordination number and particle shape. During the mechanical alloying process, the variation in geometrical characteristics of powders provides critical information. Ultrasonic parameters are shown to be sensitive not only to the average contact number per bead (i.e. the coordination number) but also to characteristics of the bead size distribution, when given the same sample preparation and confining pressure. These parameters, in turn, are sensitive to both the granular medium polydispersity and particle shapes. A non-monotonic behavior of the ultrasonic velocity (and of the derived compressional wave modulus) is observed throughout the alloying process, which thus offers possibilities for powder structure monitoring.

Contents

1. Introduction

In modern industrial applications involving metals, many research studies have been conducted in order to replace conventional materials by new structural materials exhibiting specific mechanical properties. Based on the use of a metal powder, sintering and mechanical alloying are among the fastest developing processes [1–3]. Process quality and reproducibility are highly dependent on the characteristics of the selected metal powder, which is composed of metal grains ranging in size from a few micrometers to several hundred micrometers [4]. Consequently, to improve both quality and process, it is essential to characterize in great detail the metal powder properties affecting the alloying process. The most potentially important characteristics include: morphology (average grain size, grain size distribution, grain

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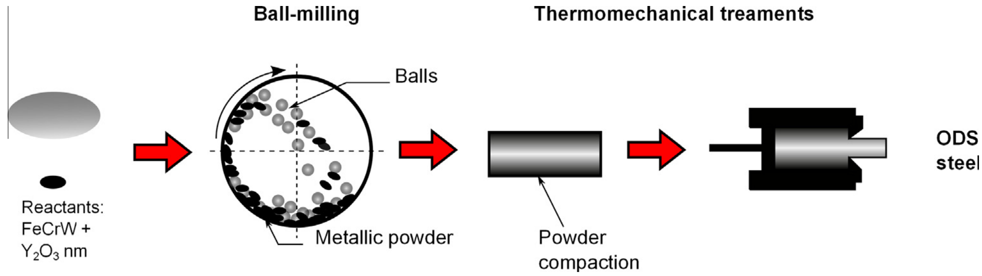

Fig. 1. Conventional process of ODS steel mechanical alloying by ball milling.

shape, etc.); the chemical, physical and mechanical properties of individual grains; and the mechanical properties of the grain packing itself. These latter properties have recently been studied using ultrasonic methods [5–8], which are known to be directly sensitive to the elastic properties of the packing configuration, through for example evaluating the ultrasonic wave velocity.

In the mechanical alloying process (described in the following Section 2), morphological characteristics represent a key piece of information on the process step [3]. In this context, it is thus desirable to implement a non-invasive method that does not require interrupting or completely halting the process (i.e. in situ probing) while at the same time provides a relatively fast and robust characterization. To facilitate implementation of ultrasonic characterization methods for this purpose, the present article will report on a study of ultrasonic wave sensitivity to the morphological characteristics of metal powders extracted at various stages of a mechanical alloying process that obviously features different properties. A laboratory-scale method of non-destructive testing will be proposed, along with a specific preparation protocol for the powder sample that is compatible with an in situ implementation. The results obtained and their interpretations demonstrate good sensitivity to the considered mechanical alloying process step. More specifically, they reveal a non-monotonic milling-time dependence for the elastic parameters associated with powder packing, which provides an initial step to quantitative characterization. This study also provides experimental results for different granular samples, and these findings may prove helpful in developing more advanced and effective medium theories regarding granular media elasticity.

2. Mechanical alloying and ball-milling process

Oxide Dispersion-Strengthened (ODS) steels have received increased interest within the nuclear energy community. It should be noted that temperatures and neutron doses in the cores of Generation IV and fusion nuclear reactors will be higher than those in Generation II and III reactors. To identify engineering solutions under such severe conditions, reliable new structural materials are needed. ODS steels are among the most promising materials in this field, thanks to a ferritic or martensitic (F/M) matrix containing dispersed nano-oxides, which exhibit low swelling under irradiation and improved creep properties at high temperature [1,2].

The standard process for creating an ODS material consists of several stages (see Fig. 1) [1,2]. First of all, Y_2O_3 and pre-alloyed powder are ground using a ball mill. Next, thermomechanical treatments are performed, according to Fig. 1. Ball milling allows Y_2O_3 to dissolve into the metal matrix and thereafter nano-oxides are formed during thermomechanical treatments. This first stage is carried out by means of a mechanical alloying process (MA). MA is a solid-state powder processing technique involving repeated welding, fracturing and re-welding of powder particles in a high-energy ball mill [3]. This technique is feasible thanks to several types of mills, including ball milling, which makes use of a cylindrical tank whose rotation around its main axis generates the milling process, as performed by steel beads existing within the powder. The grinding stems from various combinations of collisions between beads and powders on the tank walls. The powder characteristics, such as grain size or shape, evolve over the course of this

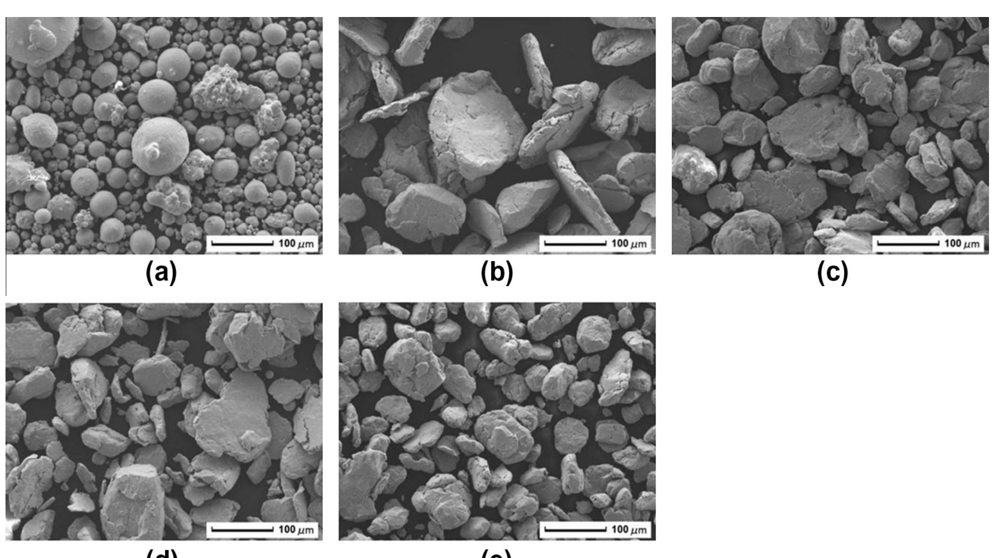

Fig. 2. Pictures obtained with scanning electron microscopy (SEM) at different mechanical alloying stages, (a) at the beginning of grinding, (b) after 44 h of milling, (c) after 88 h of milling, (d) after 176 h of milling and (e) after 352 h of milling.