Vertically-Driven Very High Speed Planetary Milling Apparatus for the Fabrication of Oxide Dispersion Strengthen Steel

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1. Introduction

The oxide dispersion strengthened (ODS) alloys are one of the most important materials for high temperature uses due to the addition of extremely thermally stable oxide particle dispersion into the matrix. Therefore, ODS alloys are the most promising materials with a potential to be applied in fast reactor fuel cladding [1,2] and fusion reactor materials [3,4]. These alloys can be widely synthesized by mechanical alloying (MA) process [5-7], which is a representative powder metallurgical technique, since any desired combination of matrix composition and dispersoid is possible.

Nevertheless, MA ODS alloys are not extensively applied in industrial fields yet because of some limitations to their use. Among their shortcomings, the relatively long time required for their production and high cost are the most important factors to be overcome. It is generally accepted that the MA process is very complicated and a very long time is needed to finish the alloying because a relatively lower milling speed is employed. For example, in order to produce MA ODS alloys, a planetary mechanical milling process is widely used at milling speeds ranging from 200 to 500rpm on the basis of supporting disk rpm. Moreover, very expensive nano-sized oxide powders should be used as starting oxide strengtheners. As a result, the use of MA ODS alloys in industry is limited even though they were developed as early as the 1960’s. In this respect, very high speed planetary milling equipment would be beneficial not only to promote the alloying process, but also to pulverize the initial coarse oxide strengtheners. In our previous works [8,9], a horizontally-driven very high speed planetary ball mill apparatus was designed and successfully applied for the fabrication of Fe based ODS alloy and nano-sized ceramic particle. However, in this horizontally-driven milling system, gravity keeps the product away from the center of the drive shaft. On the other hand, gravity effects in that way that the apparent powder concentration during processing inside the container is increasing with the distance from the top. As a result, disadvantageous dead-zone and caking effects is shown. Therefore, in order to get rid of this gravity effect, we specially designed vertically-driven very high speed planetary milling system for the fabrication of ODS alloys.

2. Methods and Results

2.1 Vertically-driven very high speed planetary ball milling apparatus

To fabricate MA ODS alloys, commercial high energy milling machines, such as Pulversette, Attritor and Spex mixer mills are often employed. Although some MA ODS alloys have been successfully fabricated using these machines, they have serious shortcomings, such as very slow alloying rate among the elements and an occurrence of dead-zone. These are caused by employment of a relatively lower rotation speed and an effect of gravity during processing, respectively.

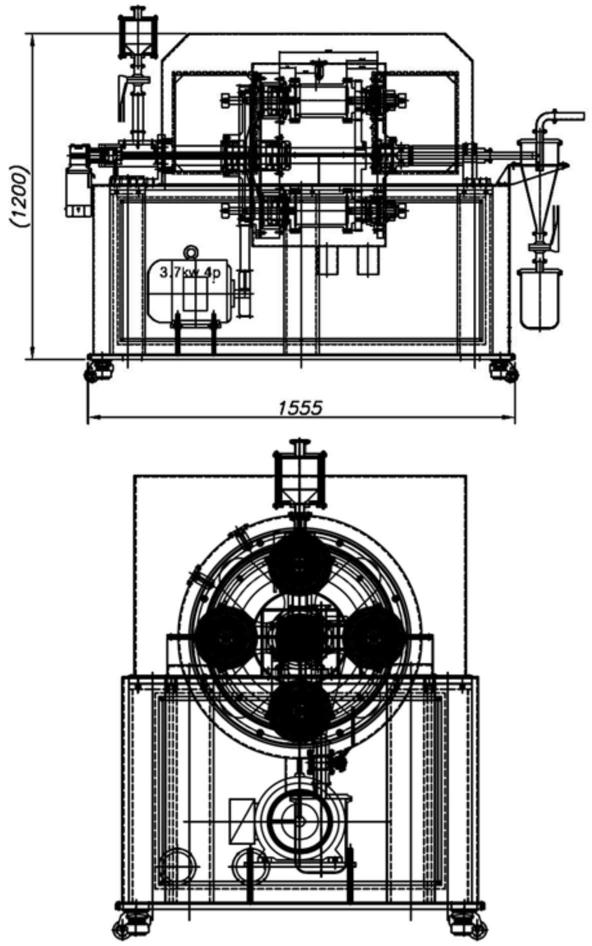


Fig. 1. Front and side drawings of vertically-driven very high speed planetary milling apparatus developed in KAERI.

To address these issues, we have designed and manufactured a new planetary milling apparatus with a very high rotating speed, which is driven vertically direction as shown in Fig. 1. The rotational speed of the vials can be raised up to a maximum of 1440rpm, which is equivalent to the centrifugal acceleration of 80G, and hence impact energy of the grinding balls can also be increased drastically compared to that in a conventional ball mill machine. The cooling circuit was laid out inside the container including both the rotating disk and vial to decrease the temperature by passing just once way. Based on these concepts, we manufactured milling apparatus as given in Fig. 2.

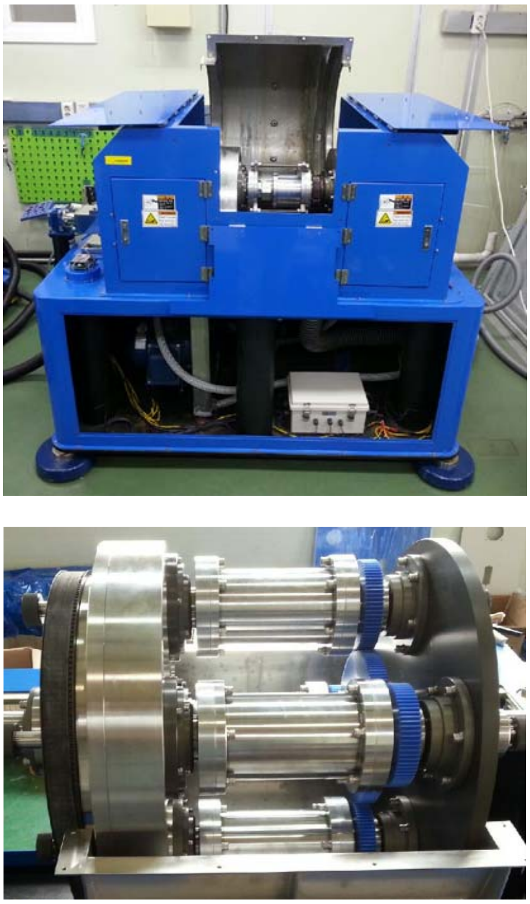


Fig. 2. Photos of entire outer and inside milling device of vertically-driven very high speed planetary milling apparatus developed in KAERI.

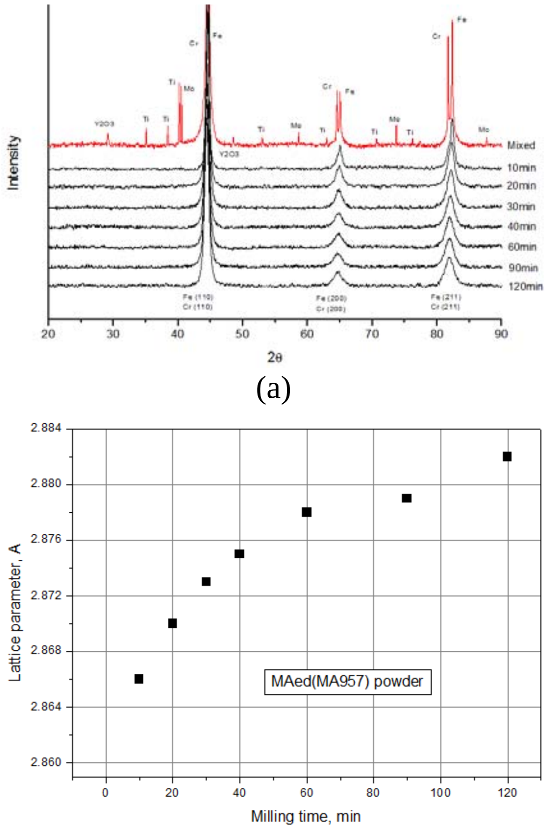
2.2 Experimental procedure

To ensure the milling ability of a new apparatus, Fe-based ODS alloy, MA957 was employed. The chemical composition was 84Fe-14Cr-1Ti-0.3Mo-0.25Y_2O_3 and Y_2O_3 oxide powder with average particle diameters of 50μm was used. Mechanical milling was conducted using our newly designed vertically-driven planetary ball mill machine under an argon atmosphere for various milling times ranging from 10 to 120mins at the rotating disk speed of 500rpm. The weight ratio of the stainless steel balls of 8mm diameter to the powder mixture was 20:1. Milled powders were mounted with acryl resin and polished in order to observe the cross-sections.

The phase the milled powders were examined by X-ray diffraction (XRD, Rigaku D/MAX 2500H) using Cu Kα radiation operating at 40kV and 100mA. The cross sections of the powder were characterized by scanning electron microscopy (SEM, FEI Sirion).

2.3 Microstructure of mechanical alloyed powders

Fig. 3(a) shows XRD patterns of MA957 ODS powders milled for various times ranging from 10 to 120min. In the XRD patterns of the initial mixed powder, c-Y_2O_3 peaks were readily observed. On the other hand, it is obvious that the XRD peaks of the Y_2O_3 particles disappeared after mechanical milling. From the XRD patterns, the full width at half maximum (FWHM) and 2theta were determined with different crystal planes for the Fe and Cr elements as a function of mechanical milling time. Then, the lattice parameter of the Fe element was calculated for the various milling times based on the Nelson-Riley relation.



(a)

(b)

Fig. 3. (a) XRD patterns of MA957 ODS powders and (b) plots of Fe lattice parameter milled for various times from 10 to 120 min.

Fig. 3(b) plots calculated lattice parameter of Fe with milling time. Considering that the value of lattice parameter of Fe was saturated after milling for 60min, it is inferred that mechanical alloying between the Fe and Cr element was finished at a milling time of 60min.

Cross-sectional backscattered SEM images of MA957 ODS powders prepared under various milling times ranging from 10 to 120min are shown in Fig. 4. In the MA powder milled for 10min, a lamellar structure between Fe and Cr was observed because mechanical alloying was not finished yet. On the other hand, in the case of MA powder milled for 60min, Fe and Cr were fully homogenized with each other; hence, it was very hard to distinguish between them with the help of SEM. In addition, the fact that there were no individual oxide particles from the cross-sectional image, suggests that the initial coarse Y_2O_3 particles became much finer and, at the same time, alloyed thoroughly with Fe and Cr. It is thus concluded that MA957 ODS powders with a higher degree of homogeneity can be achieved after milling for 60min by using a vertically-driven very high speed milling process.

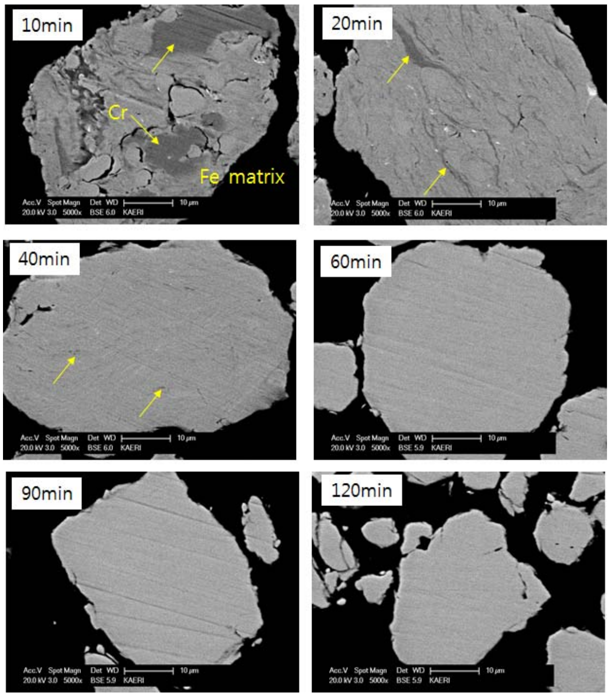


Fig. 4. Cross-sectional backscattered SEM images of MA957 ODS powder milled various milling times of 10 to 120min.

3. Conclusions

In the present work, we have designed and fabricated a vertically-driven planetary milling machine to synthesize ODS alloys. The new ball mill apparatus is able to operate under a very high rotating speed in a vertical milling direction. From analysis of the XRD patterns and the HRTEM images of the MA powders, it was found that the initial coarse Y2O3 particles readily became much finer and then thoroughly homogenized with the metal powder element in a very short milling time of 60min..

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