Formation of Rod-like Al_2TiO_5 via Mechanical Activation Followed by Thermal Processing

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Abstract

A QM-ISP-4 Planetary Mill was employed to activate mechanically the mixtures of anatase and corundum at room temperature for different times. The milled powder mixtures were then sintered at 1300 ℃ for 1 h. The XRD results showed that the milled powder mixtures were completely transformed into Al_2TiO_5 after sintering, except the mixtures milled for 5 and 10 hours. The SEM observations showed the typical morphology of rod-like Al_2TiO_5 vary in the range: widths from 0.6 to 1.2 μm, and lengths from 3.0 to 6.0 μm. The rod-like Al_2TiO_5 formation was attributed to the positive effects caused by the mechanical activation.

Keywords : aluminum titanate, rod-like crystal, mechanical activation, X-ray diffraction

1. Introduction

Al_2TiO_5 is an important engineering ceramic material due to its excellent thermal properties[1]. However, this ceramic has been restricted as a structural material due to anisotropic thermal expansion and attendant formation of microcracks which considerably reduce its strength. Many investigations have shown that the formation of rod-like crystals, which favor crack deflection and/or bridging [2], in equiaxed–grain ceramic matrix is a promising way of obtaining high fracture toughness with fairly good mechanical properties [3].

Many ceramic powders have been obtained using mechanical activation technique. This technique has advantages to use low-cost and widely available starting materials and skip the calcination, leading to a simplified process [4].

The formation of Al_2TiO_5 has not yet been studied in detail by mechanical activation. In the present work, rod-like Al_2TiO_5 have been synthesized via mechanical activation followed by thermal treatment. The preliminary experimental results on the rod-like crystals of Al_2TiO_5 will present. The effect of mechanical activation on the rod-like Al_2TiO_5 formation is discussed.

2. Experimental and Results

Al_2O_3 and anatase titania powders were used as the starting materials with the nominal composition of Al_2TiO_5.The milling operation was carried out in a planetary ball milling system in air at room temperature. Stainless steel vial and balls were used as milling media.The ball to powder mess ratio was 30:1. Powder X-ray diffraction analysis was used to determine the crystalline phases in the milled and the calcined samples. The milled powders were calcined for 1 h at 1300℃ in air atmosphere. The phase composition of the calcined samples was characterized using XRD. The microstructure and grain of the calcined samples were observed using a HITACHI S-3500 type scanning electron microscope.

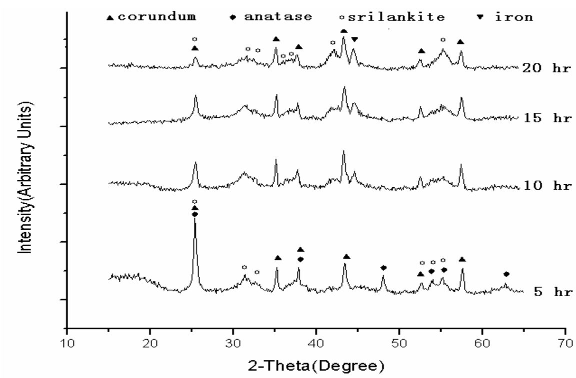

Fig. 1. XRD patterns of the milled mixture after different times of milling.

Fig. 1 shows the XRD patterns of samples ball-milled for various lengths of time. The intensity of diffraction peaks corresponding to anatase-TiO_2 decreased rapidly and those peaks of corundum became broad and weak gradually. Many reflections of anatase-TiO_2 disappeared after milling for 10 hours, at the same time, some halo-like broad peaks were clearly observed. The decrease of the intensity of the anatase-TiO_2 reflections is partially caused by the transformation of some crystalline TiO_2 to the amorphous phase and partially due to the transformation of anatase to high-pressure srilankite (TiO_2-II) phase [5].

It is evident that almost all the reflections are very much broadened as well as partially or completely overlapped. This suggests that the particle sizes of all the phases are nanometric and particles are highly strained.

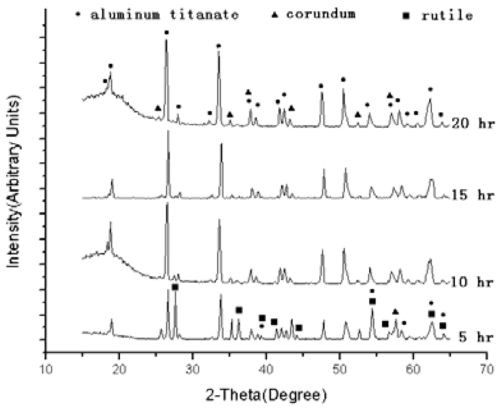

Fig. 2. XRD patterns of the calcined samples at 1300 ℃ for 1 h with different milling times.

Fig. 2 shows the XRD patterns of the milled mixtures calcined at 1300℃ for 1 h. With the increasing of milling duration, Al_2TiO_5 becomes the only predominant crystalline phase and the amount of rutile phase decreases gradually. For the sample milled for 20 h, it is very difficult to detect rutile and iron phases except predominant Al_2TiO_5 and minor corundum phases. The analyses of Fig. 3 demonstrate that nearly single phase of Al_2TiO_5 can be synthesized as the milling time duration increases to 20 h and mechanochemical processing during mechanical activation can promote the formation of Al_2TiO_5 from the ball-milled mixture.

Fig. 3 show the SEM micrograph of the calcination products. It is obvious that rod-like shape crystals are already formed in the samples sintered at 1300℃, although a few irregular shape crystals are still observed. The size distribution of the rod-like crystals becomes narrow as the ball-milling duration further increases to 20 h. The rod-like crystals presented typical dimensions varying in the following ranges: widths from ~ 0.6 to ~ 1.2 μm, and lengths from ~ 3.0 to ~ 6.0 μm.

Due to its anisotropic structure, Al_2TiO_5 might have a strong tendency to grow anisotropically if the growth process occurs under an unrestrictive environment. The formation of Al_2TiO_5 through a solid-state reaction is a diffusion-controlled procedure accompanied by an increase in molar volume of about 11%. The particle sizes reduction, phase transformation, increase of lattice strain and introduction of iron contamination simultaneously caused by mechanical activation can greatly accelerate the diffusion speed and promote the complete formation of Al_2TiO_5. The loose activated powder state during calcination is expected to provide an unconstrained environment. Therefore, the Al_2TiO_5 crystals can grow freely and the formation of rod-like crystals in this case can be achieved.

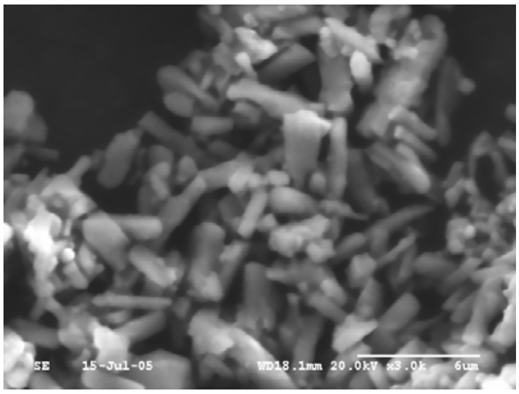

Fig. 3. SEM image of the calcined powders(milled for 20 h).

3. Summary

Rod-like crystals of Al_2TiO_5 were synthesized in mixture of corundum and anatase, via a mechanical activation process. The mechanical activation reduced the milled particle sizes, increased the lattice strains greatly, triggered TiO_2 phases transformation and introduced iron into the mixture. All the changes facilitated the formation of Al_2TiO_5 through a solid state reaction. Therefore, the Al_2TiO_5 crystal can grow freely based on its anisotropic cell structure and the formation of rod-like crystals in this case can be achieved.

4. References

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