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Collision Synthesis of Fe-based Complex Oxides with High Temperature and High Pressure Phase by High-Speed Ball-Milling

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1. Research Background

Mechanochemical grinding of powder with micro-order in diameter is suitable to be fine by general planetary ball-milling process (Ohara et al., 2008). The centrifugation with high gravity such as 150 G can realize the high collision between steal balls, which is mainly composed of shear stress between them, induced by high-speed ball-milling. In our previous study, a mechanochemical approach inspired by the Allende meteorite (Smith et al., 1981) to produce sophisticated carbon nanomaterials is reported (Ohara et al., 2010; Tan et al., 2010). It is demonstrated that unique carbon nanostructures, including carbon nanotubes, carbon onions, and new carbon nanorings are synthesized by high-speed ball-milling of steel balls. The carbon nanorings have the diameter of several ten nanometers observed by atomic force microscope (AFM). It is considered that the gas-phase reaction takes place around the surface of steel balls under local high temperatures induced by the collision energy in ball-milling process, which results in phase separated unique carbon nanomaterials. This mechanochemical effect can be also applied to synthesis Fe-based complex oxides (Ohara et al., 2010).

In this study, ilmenite was synthesized via collision-shock by high-energy ball-milling process and the existence of ilmenite with high temperature and high pressure phase was clarified from analytical measurement based on the diffraction patterns: selected area diffraction patterns and ultrahigh-resolution images.

2. Experimental Procedure

The raw material was commercially available TiO_2 (ST-01, Ishihara Sangyo, Japan) with a mean particle size of 7 nm, which was calculated from the specific surface area (SSA). TiO_2 nanoparticles with a volume of 10 cm^3 were loaded into a 180 cm^3 cylindrical vial along with 50 cm^3 milling balls. The milling balls were commercial stainless steel balls of SUS440C with a 3mm diameter. Mechanochemical treatment was performed by a high-speed ball-milling apparatus (High-G, Kurimoto Ltd., Japan) that operated for 3 h in an air atmosphere under centrifugal forces of 150 G. The phase evolution of the milled nanoparticles was characterized by X-ray diffraction (XRD; Ultima IV, Rigaku, Japan) using Ni filtered Cu-Kalpha radiation.

3. Results and Discussion

Figure 1 shows the XRD patterns of raw powder (a), and products as-milled at 150 G for 3 h by using stainless steel balls of SUS440C (b). The patterns of Fig. 1 (a) were assigned to anatase compared to the inorganic materials database of XRD patterns (supplied by NIMS atom work). The patterns of Fig. 1 (b) were assigned almost to ilmenite except iron peaks. Generally, ilmenite is formed at more than 1200 ^{o}C by solid-state reaction (Grant et al., 1972). It was suggested that the temperature inside stainless steel pot had risen locally to more than 1200 ^{o}C via higher impact energy induced by collision between balls of SUS440C. Interestingly, high temperature and high pressure phase of Fe_2TiO_4 was co-existed with Fe-rich ilmenite ($Fe_{1.5}Ti_{0.5}O_3$) and stoichiometric ilmenite ($FeTiO_3$) near 2 theta region ranging from 32 to 34^{o}. In addition, the 2theta value of Fe_2TiO_4 was extracted from the literature (Nishio-Hamane et al., 2012).

Keywords: High-speed ball-milling, Collision synthesis, Fe-based complex oxides, High temperature and high pressure phase

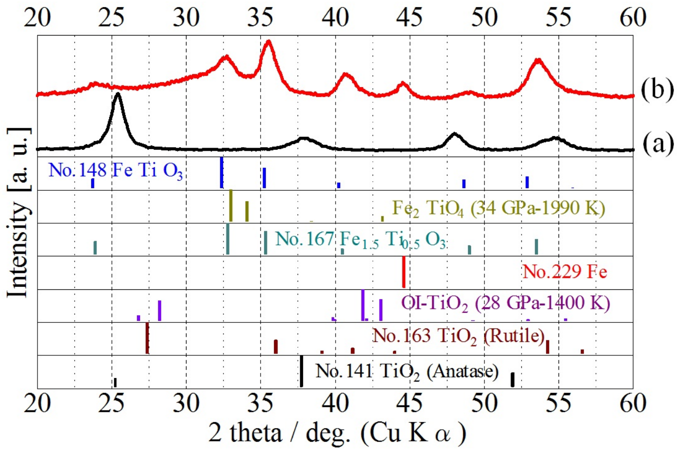

Fig.1 XRD patterns of raw powder (a) and product formed at 150 G for 3 h by using stainless steel balls of SUS440C (b).