



Repetitive grain growth behavior with increasing temperature and grain boundary roughening in a model nickel system

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Abstract

In conjunction with a previous investigation [Jung SH, Yoon DY, Kang SJL. *Acta Mater* 2013;61:5685], we have investigated the correlation between grain boundary structure and grain growth behavior in a model Ni system. Ultrafine Ni powder compacts of 180 nm size were sintered in a wide range of temperatures from 500 to 1150 °C and for various times in wet H₂. Between 500 and 600 °C, abnormal grain growth (AGG) readily occurred, with the formation of cube-shaped grains. Between 650 and 900 °C, grain growth behavior was stagnant up to 6000 min due to impingement of rapidly grown abnormal cube-shaped grains during heating. At 950 °C, some of the impinged abnormal grains suddenly grew after 20 min, showing secondary AGG behavior. As the sintering temperature was increased further, the grain growth behavior became quite normal. These changes in grain growth behavior with increasing temperature were accompanied by the structural transition of grain boundaries from fully faceted to partially faceted and defaceted. The observed grain growth behavior with respect to the grain boundary morphology is explained in terms of a coupling effect of the maximum driving force for grain growth and the critical driving force for appreciable migration of faceted boundaries. The present experimental results appear to support the validity of the previously suggested mixed control model of grain growth and the principle of microstructural evolution in polycrystals.

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Keywords: Grain growth behavior; Abnormal grain growth; Ni; Mixed control model; Microstructure evolution

1. Introduction

The structural transition of the interface in solid/liquid two-phase systems has been documented widely in the literature [1–7] following the pioneering works of Burton, Cabrera and Frank [1–3]. The coarsening behavior with respect to the interface structure, either rough (atomically disordered) or faceted (atomically ordered), has also been studied extensively for various systems [8–22]. In conjunction with these previous studies, a mixed control model of grain growth and the principle of microstructural evolution,

which reflects the coupling effect of the maximum driving force for growth (Δg_{\max}) and the critical driving force for appreciable migration of the interface (Δg_c), was suggested [23–25]. Various types of grain coarsening behavior (stagnant, abnormal, pseudo-normal and normal) were predicted as a result of the coupling effect of Δg_{\max} and Δg_c [23–25]. According to the principle, stagnant, abnormal, pseudo-normal and normal grain coarsening can take place with a reduction of Δg_c for a given Δg_{\max} . Numerous experimental results [8–17, 19–22, 26–29] support the validity of the principle for solid/liquid two-phase systems.

For single phase systems, the correlation between the boundary structure and grain growth behavior has also been studied for a number of metallic [30–36] as well as

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ceramic systems [37–45]. It has been shown that abnormal and normal grain growth occur when the grain boundary is faceted and rough [31–35,37–45]. A recent investigation on the mechanism of abnormal grain growth (AGG) in ultrafine Ni also showed that the observed AGG was related to the boundary faceting [46]. Such boundary structure-dependent grain growth behavior has, in fact, been more intensively studied in ceramic systems. In BaTiO₃, Jung et al. [43] and An and Kang [45] systematically changed the boundary structure between rough (atomically disordered) and faceted (atomically ordered) by changing the oxygen partial pressure and donor dopant concentration. They showed that the boundary structure was largely governed by the estimated total vacancy concentration – faceted and rough for low and high vacancy concentration, respectively – irrespective of the oxygen partial pressure and donor concentration. They also found that the grain growth behavior changed from stagnant to abnormal, and pseudo-normal with a reduction of the fraction of faceted boundaries, i.e. reducing the step free energy of the boundary [43]. The experimental results were well explained in terms of a coupling effect of Δg_c and Δg_{max} , as in the case of two-phase systems.

Considering previous studies in BaTiO₃ with respect to oxygen partial pressure and donor concentration [43,45], it would be worthwhile to study the boundary structure and grain growth behavior with respect to temperature to test the validity of the microstructural evolution principle in single-phase systems. Ultrafine-grained high-purity Ni was taken as a model system because grain growth occurred extensively in a wide temperature range, between 500 and 1150 °C, in this system. Grain growth behavior as well as grain boundary structure were observed at intervals of 50 °C. Various types of grain growth behavior were observed from primary stagnant grain growth (SGG) to primary AGG, secondary SGG, secondary AGG and pseudo-normal grain growth (PNGG) with increasing temperature. The observed variation of grain growth behavior with temperature has been explained in terms of a coupling effect of Δg_c and Δg_{max} , i.e. the principle of microstructural evolution [24,25,47].

2. Experimental

Commercial ultrafine Ni powder (99.9 wt.% purity, 180 nm size, JFE Mineral Ltd., Tokyo, Japan) was used to prepare samples. The powder was granulated by passing it through a 100 μ m sieve and lightly pressed into disks (9 mm in diameter and 4 mm in thickness) using a steel die. The disks were isostatically compressed at 200 MPa for 10 min using a cold isostatic press (Autoclave Engineers Inc., Erie, PA, USA). The Ni compacts were sintered at various temperatures (500–1150 °C) under flowing wet H₂ by placing them in a vertical tube furnace with one side capped. The oxygen partial pressure (P_{O_2}) of the gas was lower than the equilibrium P_{O_2} of oxidation, as noted in our previous work [46]. The heating rate of the samples

for sintering was ~ 30 °C min⁻¹. After holding the samples for various periods of time from 0 to 6000 min, they were pulled out of the furnace at a rate of ~ 30 °C min⁻¹.

The microstructures of the sintered samples were observed under both an optical microscope and a scanning electron microscope (SEM: Model XL 30S FEG, Philips, Eindhoven, The Netherlands). For the observations, the sintered samples were vertically cut and polished to a 1 μ m finish, and chemically etched with a mixed solution of 10 ml of acetic acid, 5 ml of nitric acid and 0–3 ml of distilled water. The average grain size and grain size distribution on a two-dimensional (2-D) cross-section were obtained by measuring the mean Feret diameters of grains using an image analysis program (Matrox Inspector 2.1). More than 200 grains were examined for the analysis.

Grain boundary morphologies of the samples sintered at various temperatures were observed under a transmission electron microscope (TEM: JEM3010, JEOL, Tokyo, Japan). For the observations, samples sintered at various temperatures were water-quenched to retain the grain boundary morphology at the given temperature. The water-quenched samples were ultrasonically cut into 3 mm disks, mechanically ground to a thickness of 100 μ m, dimpled to a thickness of less than 5 μ m and finally ion-milled for electron transparency. The average dihedral angle and dihedral angle distribution of the water-quenched Ni samples were measured using an image analysis program (Leica Application Suite 3.7). More than 200 junctions were examined for the analysis.

3. Experimental results

Fig. 1 summarizes the observed grain growth behaviors of UFG Ni compacts sintered in wet H₂ for various times and at temperatures between 500 and 1150 °C. As summarized in Fig. 1, various types of grain growth behavior are observed with sintering temperature and sintering time.

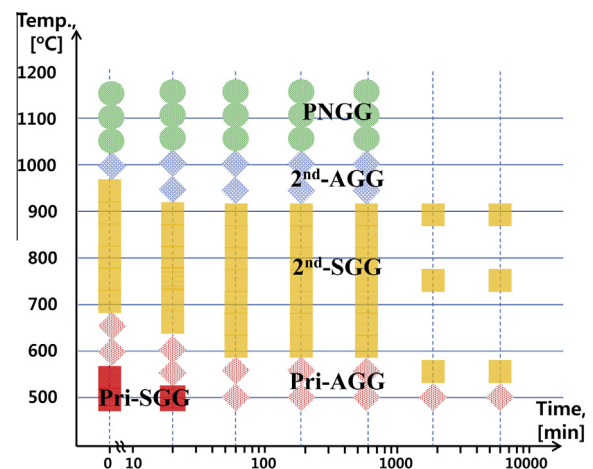


Fig. 1. Summary of the observed grain growth behavior in ultrafine Ni powder compacts at temperatures ranging from 500 to 1150 °C and for times ranging from 0 to 6000 min in wet H₂.

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