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High-Temperature Tensile Properties of a Cobalt-Based Co-20Cr-15W-10Ni Superalloy with a Bimodal Grain Structure

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Abstract: Cobalt-based superalloys are common materials for the manufacturing of various components used in aerospace applications. Conventional cobalt-based superalloys with a unimodal grain structure generally exhibit low strength and ductility at high temperatures. A bimodal grain structure of a cobalt-based superalloy, Co-20Cr-15W-10Ni (CCWN), was designed to achieve both high strength and ductility at high temperatures. The deformation behavior and tensile properties of a CCWN alloy with unimodal fine-grain (FG), coarse-grain (CG), and bimodal (FG/CG) structures were investigated at 900 °C. The microstructures and substructures after high-temperature deformation were examined via electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI) to determine the deformation mechanisms. The microstructural observation showed that the bimodal grain structure consisted of FG and CG domains. During high-temperature deformation at 900 °C, the FG structure was mainly deformed by dynamic recrystallization (DRX), maintaining a similar FG structure. The CG structure was mainly deformed by DRV, resulting in a small amount of DRX grains and a large amount of dynamic recovery (DRV) grains. However, the bimodal grain structures were mainly softened via DRX and transformed into a new bimodal structure, ultrafine grain (UFG) and FG. The FG domains tended to deform via dislocations, and the CG domains via twinning. The high-temperature tensile tests revealed that the bimodal-structured alloy exhibited both higher strength and ductility than those of the alloy samples with unimodal FG or CG structure. This is associated with the newly developed UFG/FG structures in the bimodal grain-structured samples during high-temperature deformation. This work may provide new insight into the development of high-temperature alloys with bimodal grain structures.



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Keywords: bimodal grain structure; high-temperature properties; cobalt-based superalloy; EBSD; ECCI; DRX

1. Introduction

Cobalt-based superalloys are widely used as turbine blades and inlet guide vane materials in industrial and aircraft turbines. Their stability and high strength at elevated temperatures are beneficial in the design of blades or combustion chambers in gas turbines [1–5]. The Co-20Cr-15W-10Ni (CCWN) alloy is a wrought, cobalt-based superalloy that exhibits excellent high-temperature strength, good ductility, and good corrosion resistance. Examples of applications include aerospace commercial gas turbine engines, fasteners, and wear pads [6].

The mechanical properties of the CCWN alloy at room temperature have been studied in several studies, including the effect of microstructural characteristics, such as grain size [7–12], precipitations [9,13–15], and strain-induced phase transformation [12,16–18]. Grain size strengthening is generally based on the Hall–Petch relationship, which is also valid for the CCWN alloy [7,12]. Sun reported that carbides improved the tensile properties of the CCWN alloy when the grain size was smaller than approximately 30 µm [15]. Ueki reported that a low-temperature heat treatment for the CCWN alloy could result in suppression of the strain-induced martensitic γ-to-ε transformation, thereby improving the

ductility [12,16]. Most of these studies on microstructure and mechanical properties of this alloy were conducted based on the potential biomedical applications, i.e., vascular stents, because of its outstanding mechanical properties and high corrosion resistance.

However, high-temperature deformation behavior and high-temperature mechanical properties of the CCWN alloy have been studied less, especially for high-temperature applications. Favre et al. [19,20] and Kumar et al. [21] investigated the deformation behavior and dynamic recrystallization behavior of the CCWN alloy through a high-temperature compression test. They reported that significant grain refinement occurred via dynamic recrystallization (DRX) for high temperatures and low strain rates, and DRX is the predominant mechanism occurring during hot deformation in this alloy. Gupta et al. investigated the effect of microstructure (grain size) on mechanical properties of the CCWN alloy [22]. They reported that the room-temperature strength of the alloy depended on the grain size; however, a strength at 980 °C was independent of the grain size. Knezevic et al. investigated the deformation behavior of the CCWN alloy at a temperature range of 100–400 °C [6]. They reported that planar glide dominated the process of plastic deformation in this alloy in this temperature range. Zafarghandi et al. studied the effects of Nb on the hot tensile deformation behavior of the cast CCWN alloy in a temperature range of 950 to 1200 °C, and demonstrated that dynamic recovery (DRV) is the main deformation mechanism when the deformation temperature is lower than 1000 °C [23]. Similarly, Chiba et al. revealed that deformation twinning can occur in Co-Ni-Cr-Mo (SPRON510) Co-Ni-based superalloys during high-temperature deformation, as a result of dynamic strain aging [24]. The previous studies mainly focused on the deformation mechanisms of the CCWN alloy at elevated temperatures. The effect of microstructures, such as grain size and its distribution, on the high-temperature properties of the CCWN alloy is not as clear as at room temperature.

Our previous studies revealed that the CCWN alloy with a bimodal structure that consisted of fine grains (FGs) and coarse grains (CGs) exhibited high strength and ductility at room temperature [25–28]. A recent study found that the bimodal grain structures of this alloy are also thermally stable at temperatures below 1100 °C [29]. This may indicate that the bimodal grain-structured CCWN alloy can be also used at high temperatures. However, the high-temperature mechanical performance of the bimodal grain-structured CCWN alloy remained unknown and needed further studies. Therefore, the present study focused on the high-temperature mechanical performance of the CCWN alloy with a bimodal structure. The performance of the alloy samples with unimodal grain structures of FGs and CGs was also investigated for comparison.

2. Materials and Methods

The material used in the present study was a CCWN alloy. The chemical composition of the material (in mass%) is Cr: 20.6, W: 14.9, Ni: 10.8, Fe: 2.7, Mn: 1.7, C: 0.1, and Co balance. The raw materials in the shape of bars were received from SamHwa steel Company, Busan, Korea. The as-received bars with a diameter of 12 mm were solution treated at 1250 °C for 30 min, followed by water quenching. The water-quenched bars were cold rolled at room temperature by multi-pass until there was an area reduction of 30%. The cold-rolled bars were then annealed at 1000 °C, 1100 °C, and 1200 °C for 15 min followed by water quench to generate different microstructures based on our previous studies [25,28].

The samples for microstructural observation were mechanically ground using abrasive papers 400#–2000# and then polished with 5 µm and 1.5 µm diamond paste and 0.04 µm OPS solution. Microstructure characterizations were conducted using a backscattered-electron (BSE) detector and an electron backscatter diffraction (EBSD) system in a field-emission scanning electron microscope (FESEM: TESCAN MIRA III, Brno, Czech Republic). The EBSD measurements were performed using an AZtec EBSD system operated at 20 kV. EBSD maps were taken with a working distance of 15 mm and step sizes of 0.4 and 1 µm. The grain sizes and volume fractions of FGs and CGs were calculated by using an OIM Analysis v8 program. Electron channeling contrast imaging (ECCI) was used to characterize the deformed substructures in an FESEM (Zeiss Gemini 500, Oberkochen,

Germany) operated at 20 kV and under high-current mode. A working distance of 3–4 mm and an aperture size of 60 μm were used.

The bar-type tensile samples were prepared by conventional machining and the gauge portions were ground to a surface roughness of 0.8. Tensile samples had a gauge dimension of 35 mm in length and 4 mm in diameter. Tensile tests were performed at 900 °C by using a SHIMADZU AG-IC250KN (SHIMADZU, Tokyo, Japan) machine. The test temperature was selected based on the service temperature of this alloy in gas turbine engines, which is 760–870 °C [30,31]. A constant strain rate of $2.4 \times 10^{-4} \text{ s}^{-1}$ was used, which was determined by using a contact extensometer. The samples were heated to the test temperatures at 5 °C/s and soaked for 10 min to put down any temperature gradient. The tensile tests of the samples under each condition were carried out three times for average values of the ultimate tensile strength (UTS), yield strength (YS), and elongation (El). The UTS and YS values of the samples were measured from the engineering stress–strain curves, and the El values were measured through putting the fractured samples back together.

3. Results

3.1. Initial Microstructures

Figure 1 shows the microstructures and grain-size distributions of the alloy samples. As shown in Figure 1a–a'', the as-received sample exhibited a fine-grained structure with an average grain size (d_{avg}) of 2.1 μm , and most of the grains are finer than 5 μm . Herein, the as-received sample will be referred to as FG sample. In addition, the FG sample contained carbides, as indicated by the arrows in Figure 1a. EBSD analysis confirmed that the coarse carbides with a size of a few microns were M_{23}C_6 , whereas the fine carbides formed along the grain boundaries were M_7C . Figure 1b–d shows the microstructures of the alloy samples after cold-rolling and annealing at different temperatures. The samples annealed at 1000 and 1100 °C exhibited d_{avg} values of 5.5 and 6.9 μm , respectively. However, a bimodal grain structure that consisted of considerably different fractions of FGs and CGs can be observed in the samples, as shown in Figure 1b,c. The sample annealed at 1000 °C exhibited a higher fraction of FGs (approximately 68.8%), whereas the sample annealed at 1100 °C exhibited a lower fraction of FGs (approximately 50%). Herein, the samples annealed at 1000 and 1100 °C will be referred to as FG/CG-1 and FG/CG-2, respectively. Additionally, very fine carbides on the nanoscale were observed in the samples, as indicated by the arrows in Figure 1b,c. These carbides were M_{23}C_6 and M_7C that were newly developed during annealing at 1000 and 1100 °C. The sample after annealing at 1200 °C exhibited a coarse-grained structure with a d_{avg} of 53.1 μm , as shown in Figure 1d. Most of the grains were coarser than 5 μm . Herein, the sample annealed at 1200 °C will be referred to as CG sample. In addition, no carbides were observed in the CG sample.

3.2. High-Temperature Tensile Properties

Figure 2 shows the stress–strain curves and tensile properties of the alloy samples tested at 900 °C. The FG and FG/CG samples tested at 900 °C exhibited a similar flow behavior, i.e., rapid softening occurred after yielding in these samples, as shown in Figure 2a,b. On the contrary, the CG sample exhibited a discontinuous yielding phenomenon after yielding, which was then followed by a noticeable work hardening and a gradual softening. Figure 2c indicated that in the FG and FG/CG samples, the strain hardening occurred in the beginning of deformation until a strain of approximately 0.1. The strain hardening in the CG samples occurred until a strain of approximately 0.3. Figure 2d shows the high-temperature tensile properties of the samples. At 900 °C, the CG sample exhibited the lowest strength and ductility, i.e., a UTS of $156 \pm 1 \text{ MPa}$, a YS of $154 \pm 0 \text{ MPa}$ and an El of $67.2 \pm 13.8\%$. The FG sample exhibited a UTS of $217 \pm 2 \text{ MPa}$, a YS of $204 \pm 3 \text{ MPa}$, and an El of $75.5 \pm 6.4\%$. The FG/CG samples exhibited both higher strength and ductility than those of the FG and CG samples. For instance, the FG/CG-1 sample showed a UTS of $224 \pm 2 \text{ MPa}$, a YS of $215 \pm 6 \text{ MPa}$, and an El of $91.3 \pm 1.3\%$. The FG/CG-2 sample showed very similar strength and ductility to that of the FG/CG-1 sample.

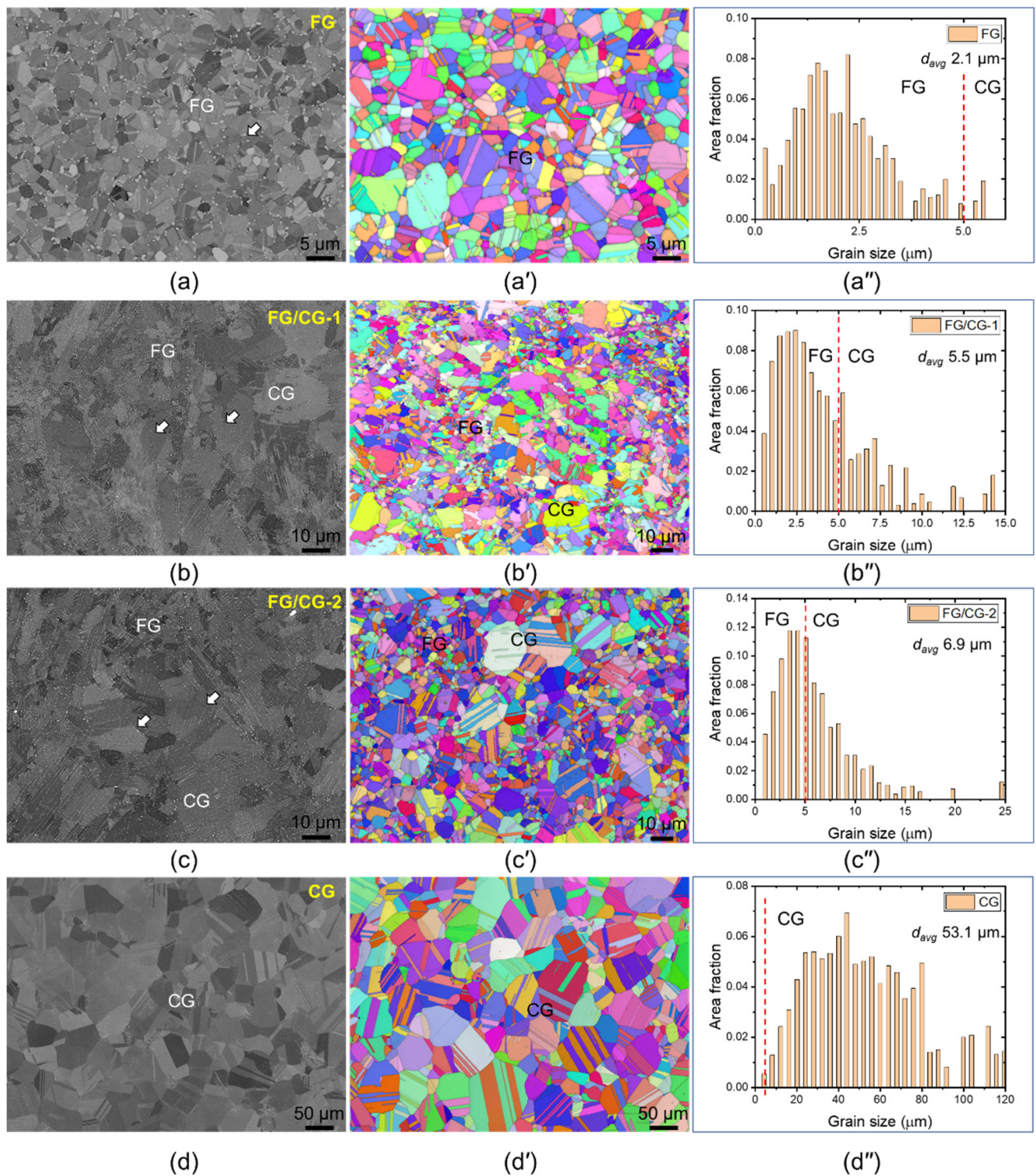


Figure 1. BSE and EBSD maps showing the microstructures of the as-received samples (a–a''), and the samples annealed at 1000 °C (b–b''), 1100 °C (c–c'') and 1200 °C (d–d'').

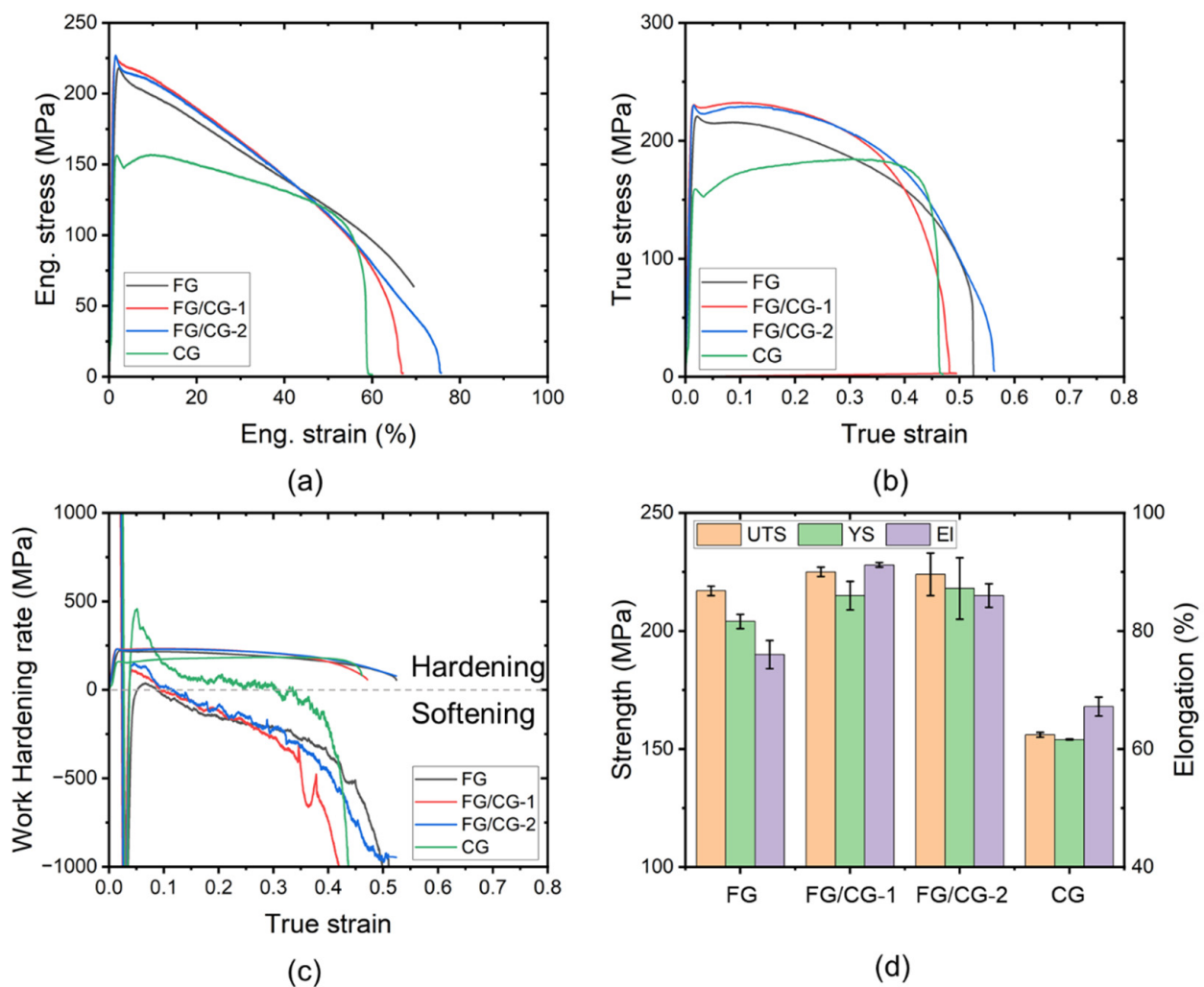


Figure 2. Tensile properties of the alloy samples tested at 900 °C: (a) engineering stress–strain curves, (b) true stress–strain curves, (c) work hardening rate, and (d) strength and elongation.

3.3. Grain Structure Evolution during High-Temperature Deformation

EBSD maps in Figure 3 show the microstructural evolution of the FG and CG deformed samples, with a strain level of approximately 20%. It is observed that the deformed FG sample exhibited fine grains, whereas the deformed CG sample still had very large grains, as shown in Figure 3a,d. DRX occurred in the FG sample, with a fraction of approximately 65.6% based on the grain orientation spread (GOS) map in Figure 3b. DRX grains and deformed grains were identified based on if their GOS was below or above 2° , respectively. On the contrary, very few DRX grains were observed in the CG samples, resulting in a DRX fraction of 16.7%. Grain size maps in Figure 3c,f indicate that the deformed FG sample exhibited a d_{avg} of 2.4 μm , which is like that of the initial FG sample. The d_{avg} of the CG sample decreased from 50.3 to 12.5 μm after deformation of 20%. In addition, the deformed FG sample contained a considerable amount of ultrafine grains (UFGs) with a fraction of 13.7%, as shown in Figure 3c. The formation of the UFGs is due to dynamic recrystallization. This indicated that the FG sample after deformation exhibited a new bimodal structure consisting of UFGs and FGs. On the contrary, the CG sample had a very small number of UFGs, only 5.3%; the CG sample then consisted of FGs (a fraction of 26.1%) and CGs (a fraction of 68.6%), as shown in Figure 3d.

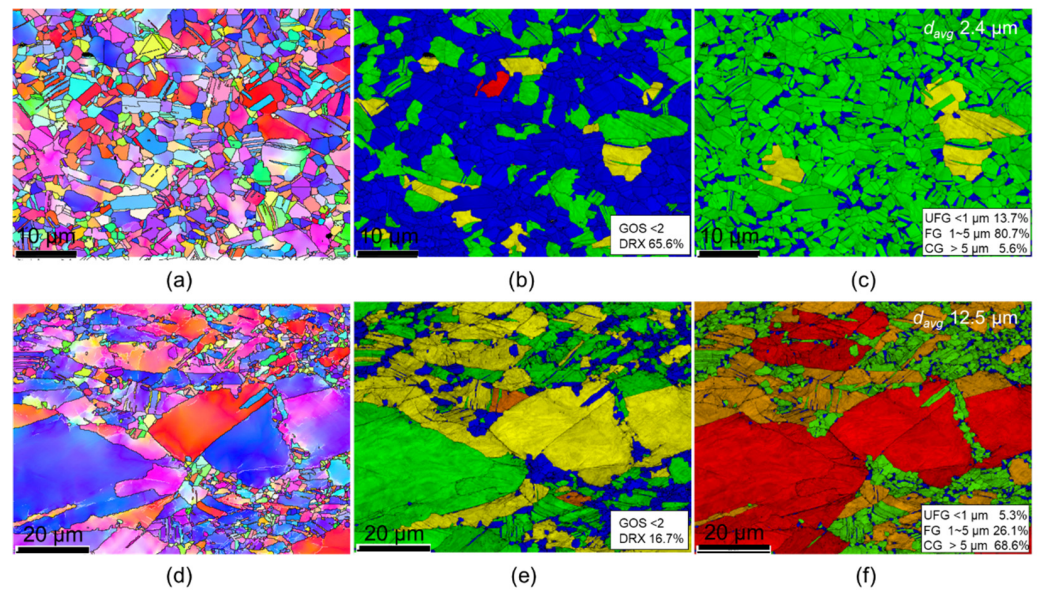


Figure 3. EBSD-IPF (a,d), grain orientation spread (GOS) (b,e), and grain size (d,f) maps of the FG (a–c) and CG (d–f) samples deformed at a strain of 20%.

EBSD maps in Figure 4 show the microstructure of the deformed FG/CG samples. It is observed that the bimodal structure consisting of both FGs and CGs remained in the deformed samples, as shown in Figure 4a,d. The GOS maps in Figure 4b,e revealed that the FG/CG-1 had a larger fraction of DRX, approximately 76.8%, and the FG/CG-2 sample exhibited a DRX fraction of 52.6% because of the higher fraction of CGs in the initial sample. The grain size maps in Figure 4c,f showed that the d_{avg} of the FG/CG-1 and FG/CG-2 samples decreased from 5.5 to 1.7 μm and 6.9 to 2.2 μm , respectively. In addition, the samples contained approximately 20% of UFGs and 70% of FGs. This indicated that the FG/CG samples after deformation developed a new bimodal structure, which consisted of UFGs and FGs.

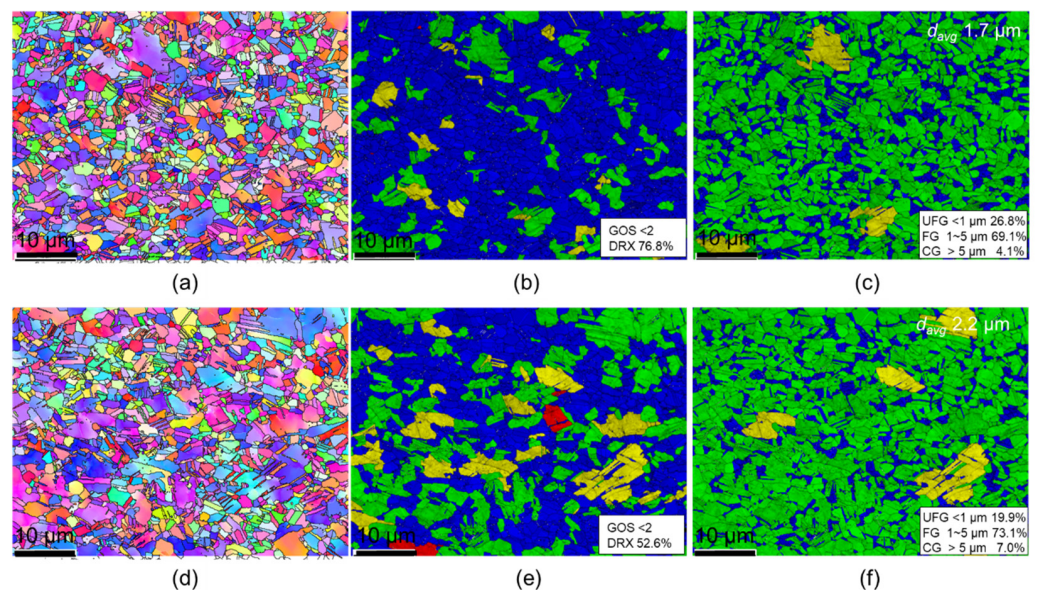


Figure 4. EBSD-IPF (a,d), GOS (b,e), and grain size (d,f) maps of the FG/CG-1 (a–c) and FG/CG-2 (d–f) samples deformed at a strain of 20%.

3.4. Substructure Evolution during High-Temperature Deformation

ECC images shown in Figure 5 reveal the substructural evolution of the FG and CG samples during deformation. In the deformed FG sample, the previous grains with a coarser size tended to deform through deformation twinning, as indicated by an arrow in Figure 5a. The finer grains were deformed through dislocations, and a high density of dislocations was observed, as shown in Figure 5b. In the CG sample deformed at 900 °C, the previous grains were elongated along the tensile direction, and only a few DRX grains can be observed at the previous grain boundaries. Both dislocations and deformation twins were frequently observed in the deformed grains, as shown in Figure 5c,d.

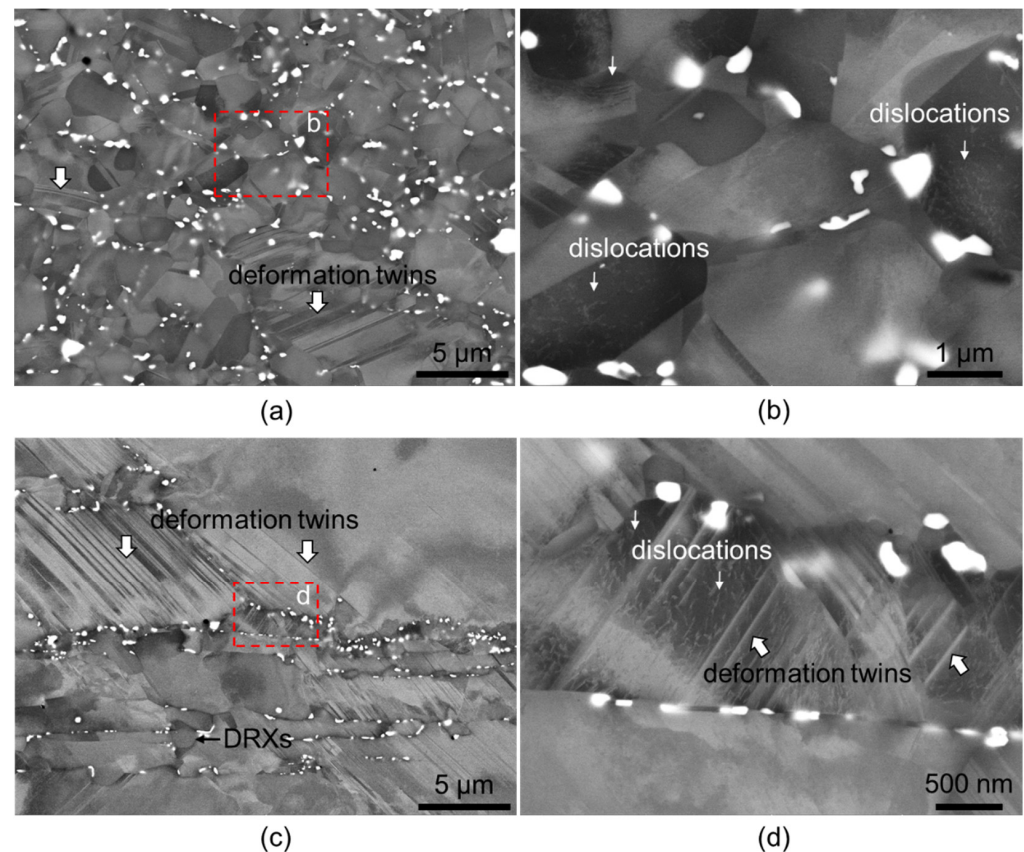


Figure 5. ECC images showing the substructures of the deformed samples: (a,b) FG and (c,d) CG.

Figure 6 shows the substructures of the deformed FG/CG samples. It is observed that the FG/CG-1 sample was mainly deformed through dislocations and DRX, as shown in Figure 6a. Only a few deformation twins are observed, as shown in Figure 6b. In addition, the DRX also induced the formation of UFGs, as shown in Figure 6b. In the FG/CG-2 sample, the CGs were deformed through dislocation and twinning. In addition, the previous FGs were transformed into UFGs through DRX.

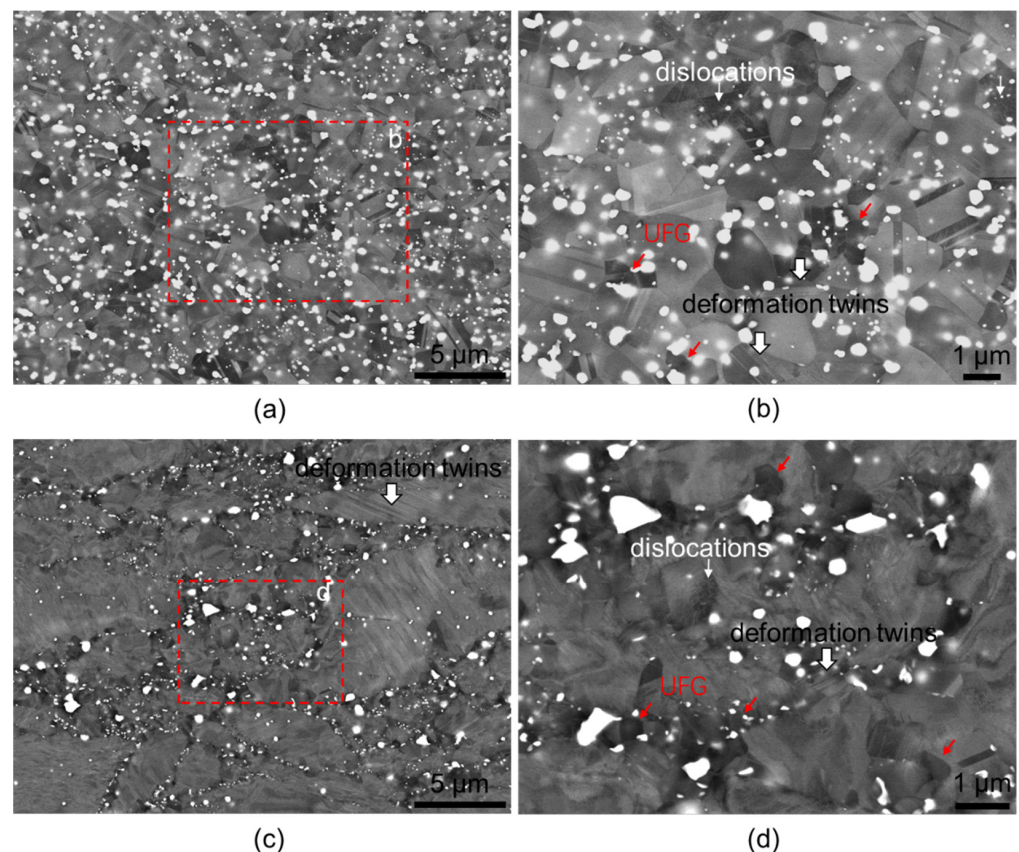


Figure 6. ECC images showing the substructures of the deformed samples: (a,b) FG/CG-1 and (c,d) FG/CG-2.

4. Discussion

Bimodal structures in metals and alloys have attracted attention because of their contribution to high strength and ductility. Numerous studies have reported that bimodal structure can simultaneously enhance the strength and ductility of metallic materials at room temperature, i.e., steel, light metals, and high-entropy alloys [32]. However, the mechanical performance of the bimodal structure at high temperature is unclear, especially those superalloys, such as Ni-based and Co-based alloys. This work demonstrated that the cobalt-based superalloy CCWN with a bimodal structure exhibited superior mechanical performance, both at room temperature and at more elevated temperatures than their unimodal ones. The high performance at room temperature is because the FG and CG domains in the samples have significant contributions of high strength and ductility, respectively [25,26,33]. The promising properties at high temperature are due to the dynamically developed new bimodal structure, UFG/FG, which was formed via DRX. The details are discussed below.

In general, the DRX and DRV processes occur in metals deformed at high temperature, depending on the temperature, strain rate, and deformation content [34]. Previous studies revealed that DRX is the main deformation mechanism in the CCWN alloy deformed at above 1000 °C, especially in the case of higher temperature and lower strain rate [19]. DRX is hardly observed in the CCWN alloy when deformed at temperatures below 1000 °C. For example, a recent work reported that DRX has not occurred in the CCWN alloy when deformed at 950 °C [23].

The initial microstructural characteristics, such as grain size and its distribution, also play an important role in the final microstructure during subsequent hot deformation. In this work, the unimodal FG grain structure under high-temperature deformation at 900 °C generated an FG structure through DRX. The DRX grains were deformed through

either dislocation or twinning. The grain size of the FG structure after high-temperature deformation did not change significantly. On the contrary, the CG structure under high-temperature deformation exhibited the coexistence of DRX and DRV, resulting in a large fraction of coarse DRV grains and a small fraction of fine DRX grains. This indicated that the initial grain size has a significant effect on the final microstructure after high-temperature deformation, leading to different high-temperature mechanical properties. The sample with an FG structure exhibited both higher strength and ductility than those of the one with a CG structure, as shown in Figure 2. However, the bimodal grain structure, having both FG and CG domains, coexisted in the samples. The two domains during high-temperature deformation behave differently upon high temperature: First, the FG domains tend to deform into UFGs via DRX, while the CG domains tend to deform into FGs via DRX or CGs via DRV. Second, the FG domains were deformed mainly by dislocations, whereas the CG domains were deformed by twinning. This resulted in both high strength and ductility in the bimodal grain-structured samples in comparison to the samples with an FG or a CG structure.

The findings in this work indicated that the CCWN alloy with a bimodal grain structure exhibited superior mechanical properties, both at room temperature and elevated temperature compared to a unimodal grain structure. This work may provide new insight on the development of high-temperature alloys.

5. Conclusions

The present study investigated the high-temperature deformation behavior and mechanical properties of the Co–20Cr–15W–10Ni (CCWN) alloy that had a bimodal grain structure. The alloy samples with unimodal grain structures were also investigated for comparison. The main conclusions are drawn as follows:

- (1) During high-temperature deformation at 900 °C, the FG structure was mainly deformed by DRX, maintaining a similar FG structure. The CG structure was mainly deformed by DRV, resulting in a small amount of DRX grains and a large amount of DRV grains.
- (2) The bimodal grain structures were mainly deformed via DRX and transformed into a new bimodal structure, ultrafine grain (UFG) and FG. The FG domains tended to deform via dislocations, and the CG domains via twinning.
- (3) The bimodal-structured alloy exhibited both higher strength and ductility than those of the alloy samples with unimodal FG or CG structure. This is associated with the newly developed UFG/FG structures in the bimodal grain-structured samples during high-temperature deformation.

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