Dynamic Recrystallization Behavior of Biomedical CCM Alloy with Additions of C and N

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In order to examine the dynamic recrystallization (DRX) behaviors of Co-29Cr-6Mo alloy with additions of both C and N (hereafter CCMCN alloy), uniaxial compression tests in the temperature range of 1273 to 1473 K and strain rates of 0.01 to 30 s⁻¹ were carried out. The influence of hot forging conditions (i.e., temperature, strain rate, and strain) on the microstructure of deformed sample was investigated in detail by means of electron backscattering diffraction (EBSD) and optical microscopy (OM). The results revealed that the initial microstructure is a stable γ face-centered cubic (FCC) phase with a large number of M₂₃C₆ precipitates both inside the grains and at grain boundaries. The DRXed grains were observed to be uniformly distributed and to decrease with strain. The high volume fraction of $\Sigma 3$ boundaries after the DRX was observed, indicating a close relation between the DRX mechanism and $\Sigma 3$ boundary formations. In addition, the activation energy Q of CCMCN alloy was observed to be higher as compared to those of alloys without C or N addition and that with N addition.
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1. Introduction

Co-Cr-Mo alloys have been widely used in biomedical implants such as artificial hip joint and knee joints for their excellent corrosion resistance, wear resistance, and biocompatibility. However, it is well known that Co-Cr-Mo alloys have extremely low stacking fault energy (SFE) even at high temperatures (approximately 22 mJ m⁻² at 1273 K as calculated by Yamanaka *et al.*¹⁾ in the case of Co-29Cr-6Mo alloy), because the additions of both Cr and Mo reduce the SFE at temperatures below 1273 K.²⁾ The low stability of the γ phase due to the low SFE of Co-Cr-Mo alloys greatly hampers their applications in biomedical implants and conventional structural components because they have poor elongation and workability at room temperature. Ni was thus added to improve the plasticity of this alloy. However, it has been reported that the release of Ni ions into the human body has a high possibility of resulting in an allergic reaction.³⁾ Our previous research has shown that the addition of N, C, or Zr can greatly enhance the stability of the γ phase by increasing the SFE of CCM alloy.⁴⁾ On the basis of our previous results observed using a three-dimensional atomic probe, the stabilizing effect of the γ phase by the addition of N or C is ascribed to the stable crystalline structure formed between Co-Cr-N (or C) atoms⁵⁾ because chromium has a much stronger interaction with the interstitial N or C than that which exists between Co and N (or C).

According to research by Chiba *et al.*,⁶⁾ a mean grain size of approximately 3 μ m can be obtained through hot forging at temperatures higher than 1273 K in Co-29Cr-6Mo alloy without the addition of N or C (hereafter CCM) alloy. The mechanical properties were also improved greatly by grain refinement. The dynamic recrystallization (DRX) behavior in this alloy was investigated in detail by Yamanaka *et al.*⁷⁾

Their results indicated that the DRX was related to the low SFE of this alloy and the dense planar dislocation structures due to the formation of SFs possibly inducing the grain refinement.

Basic research regarding the phase transformation behavior and mechanical properties in CCM alloy with N addition has also been carried out by Kurosu *et al.*⁸⁾ Moreover, improvements in the mechanical property of CCM alloy through the addition of C or Zr have been reported by Lee *et al.*⁹⁾ An examination of the DRX behavior of CCM alloy with addition of N was also recently conducted by Li *et al.*¹⁰⁾ in which the $\Sigma 3$ twinning boundary formation was considered to be the dominant DRX mechanism due to the low SFE in this alloy. However, the DRX behavior of CCM alloy with additions of both C and N has not yet been clarified.

In this context, the purpose of this research is to investigate the DRX behavior of a Co-29Cr-6Mo alloy with addition of both C and N (hereafter CCMCN alloy). It is widely known that the DRX behavior of alloys is strongly influenced by the working condition (i.e., strain rate, working temperature, and strain).¹¹⁾ The effects of these working conditions on the DRX behavior of CCMCN alloy will be analyzed in detail.

2. Experimental Procedure

Co-29Cr-6Mo-0.23C-0.14N alloy (mass%) was used in the current study. Cylindrical specimens, 8 mm in diameter and 12 mm in height, were machined by electro-discharge machining (EDM). To reduce the non-uniformity of the microstructure due to friction between the sample surface and the jig, the flat ends of the specimen were machined with concentric grooves of 0.1-mm depth so that the lubricant could flow freely inside the grooves in a molten state at high temperatures, resulting in a large decrease in the friction coefficient.¹²⁾ In order to avoid heat dissipation from the

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Fig. 1 (a) Configuration of the specimen during the hot compression test; (b) Schematic schedule of hot compression tests.

sample end surface to the anvil surface at the contact region, mica sheets with a diameter of 20 mm and thickness of approximately 0.2 mm were used as heat insulators and were placed in close contacting with the anvil surfaces; because of their extremely low thermal conductivity, they were placed close to the anvil surface. In addition, a graphite sheet was used as an aid to reduce friction owing to its high heat resistance and high lubricating effect (Fig. 1(a)).

Compressive tests were carried out in a vacuum from 1273 to 1473 K, increasing in steps of 50 K using a computer-aided Thermecmaster-Z hot-forging simulator. The selected strain rates were 0.01, 0.1, 1.0, 10, and 30 s⁻¹. The specimen was heated to 1473 K at a rate of 5 K s⁻¹ by high-frequency induction heating; it was then subjected to homogenizing heat treatment for 300 s after reaching the target temperature. As soon as the sample was compressed to the final strain

level, it was quenched with a mixture of N₂ (6 MPa pressure) and He (4 MPa) at a cooling rate of approximately 50 K s⁻¹ to room temperature (Fig. 1(b)).

Crystallographic analysis was conducted by electron backscattering diffraction (EBSD) using an orientation imaging microscope (TexSEM Laboratories, Inc., Provo, UT) attached to a field-emission scanning electron microscope (FE-SEM). The surface of the specimen for microstructural observation was prepared by first dry grinding it with SiC emery paper. Electrolytic polishing was subsequently conducted in a sulfuric acid-methanol (1 : 9) solution. Microstructure observation was carried out by FE-SEM and electron probe microanalysis (EPMA). Due to the large number of fine precipitates that formed in the initial microstructure of this alloy, electrolytic extraction was conducted to the alloy in 10% diluted sulfuric acid electrolyte at 6 V for 7.2 ks, and X-ray diffraction (XRD) was applied to analyze the exact structure of these precipitates.

3. Results and Discussion

3.1 Initial microstructure of CCMCN alloy

Image quality (IQ) maps obtained from the EBSD and XRD profile of the CCMCN sample before compression are shown in Figs. 2(a) and (b), respectively. The sample was annealed at 1473 K for 300 s for solution treatment followed which it was annealed at 1423 K for 300 s; rapid cooling at a rate of 50 K s⁻¹ was carried out after annealing. The initial γ grain size was assumed to be 20 to 30 μ m with extensive Σ 3 boundaries within the matrix (Fig. 2(a)). The XRD result indicates a single γ phase in the initial microstructure (Fig. 2(b)). No martensitic transformation occurred and no thermal ϵ phase existed after cooling, indicating a much more stable γ phase as compared to that in the CCM alloy without C or N addition, in which over half the γ phase transformed into ϵ phase by martensitic transformation during a similar rapid cooling process.¹³⁾ Although the results are not presented here, after various hot compression processes in the current study, minimal ϵ phase was detected by both XRD and EBSD, indicating the effective stabilization of the γ phase with the addition of C and N in all cases.

Fig. 2 Initial microstructure of CCMCN alloy after solution treatment at 1473 K for 300 s followed by annealing at 1423 K for 300 s. (a) Image quality (IQ) map and (b) X-ray diffraction (XRD) profiles.

Fig. 3 (a) Phase map of Co-29Cr-6Mo-0.23C-xN alloy in which the x-axis represents the N content; (b) phase map of Co-29Cr-6Mo-0.14N-xC alloy in which the x-axis represents the C content.

Fig. 4 (a) Field-emission scanning electron microscopy (FE-SEM) image and (b) composition mapping of C, Cr, Mo, and N elements in the as-received CCMCN alloy obtained by electron probe microanalysis (EPMA).

3.2 Identification of precipitates

Numerous precipitates in both the interior of the grains and along the grain boundaries are evident in the IQ map, as shown in Fig. 2(a). These precipitates can be observed in both the as-received sample and the sample after solution heat treatment. In order to quantitatively identify these precipitates, phase diagrams were calculated using a Thermo-Calc, as shown in Figs. 3(a) and (b). Figure 3(a) shows a phase diagram of Co-29Cr-6Mo-0.23C-xN alloy in which the x-axis indicates the contents of N elements, and Fig. 3(b) shows a phase diagram of Co-29Cr-6Mo-0.14N-xC alloy in which the x-axis indicates the contents of C elements. According to these two diagrams, there is a high possibility that the precipitates are M_2N , $M_{23}C_6$ or σ phase

because annealing treatment of this alloy was conducted from 1273 to 1473 K, and the contents of C and N are 0.23% and 0.14%, respectively. Because there is no significant change in the profiles of precipitates, the as-received sample was chosen to be the object for analysis by SEM imaging, as shown in Fig. 4(a). The precipitates are observed to be light in color. The corresponding variations of the element distribution of C, Cr, Mo, and N in this surface were studied by EPMA, as shown in Fig. 4(b). In the figures, the composition of each element increases as the color changes from blue to green and to red and pink in an arbitrary scale. It is evident from these results that the precipitates are composed of a large quantity of C, Cr, and Mo; very little N is observed to exist in these precipitates, implying that most

of the N elements resolved into the matrix, whereas a part of the C element instead resolved into precipitates along and inside the initial grains. The results of XRD analyses applied to the precipitates obtained from electrolytic extraction for the as-received sample and the sample after the same solution treatment are shown in Figs. 5(a) and (b), respectively. Most of the peaks in the XRD pattern were identified to be $M(\text{Cr}, \text{Mo})_{23}\text{C}_6$. A slight σ phase peak was also observed in both Figs. 5(a) and (b), implying that very little σ exists in this alloy throughout the hot forging process. On the basis of the above results, the main precipitates of CCMCN alloy are considered to be $M(\text{Cr}, \text{Mo})_{23}\text{C}_6$. According to the result of Oscobedo *et al.*,¹⁴ adding the N element into CCM alloy with C addition will lead to the precipitation of $M_{23}\text{C}_6$ or $M_6\text{C}$ carbides, indicating that there should be a stronger interaction between the N element and the Co, Cr, or Mo element than that in the C element at high temperature. The results of the present study are Current research is considered to be in good agreement with those of Oscobedo *et al.*¹⁴ in that the addition of both N and C give rise to the preprecipitation of a $M_{23}\text{C}_6$ compound. In this case, the phase map calculated by Thermo-calc is not in

good agreement with the actual one for this alloy. However, our basic research showed that Cr_2N is easily dissolved into the matrix at temperatures above 1323 K, whereas the carbides do not dissolve significantly even at 1473 K.⁸) This is thought to be another possible reason for the absence of Cr_2N in the current alloy because a solution treatment followed by a quick cooling was applied to the sample and the dissolved Cr_2N does not have sufficient time to precipitate.

3.3 Compressive stress–stress-strain curve

Figures 6(a) and (b) show the effect of strain rate and temperature on the true stress–true strain (σ – ϵ) curves obtained from the hot compression tests. Figure 6(a) shows the σ – ϵ curves obtained at various strain rates from 0.01 to 30 s^{-1} at a fixed deformation temperature of 1423 K. Figure 6(b) shows the results when the deformation temperature was varied at a fixed strain rate of 1 s^{-1} . For all deformation conditions, the σ – ϵ curves exhibit typical DRX characteristics; specifically, the flow stress increased to a peak stress that is followed by work softening; steady-state flow is attained at higher strains. Furthermore, all obtained σ – ϵ curves have a single peak; neither the stress oscillations in the σ – ϵ curves with multiple stress peaks nor dynamic recovery in the σ – ϵ curves in which steady-state flow develops without work softening were observed.

3.4 Effect of strain on DRX behavior

A typical IQ map of the microstructure obtained by EBSD compressed to a reduction rate of 60% at 1423 K and 1 s^{-1} is shown in Fig. 7(a). The mean grain size in Fig. 7(a) is estimated to be approximately $3.4 \mu\text{m}$. A very uniform distribution of refined grains was obtained, and no grain growth was observed with further compression. It is evident that most of the DRXed grain boundaries are characterized by the formation of $\Sigma 3$ boundaries, indicating that the DRX process in CCMCN alloy proceeds through the prolific and uniform formation of $\Sigma 3$ boundaries in the initial microstructure. The mean grain size as a function of the true strain level is shown in Fig. 7(b). It is evident that mean grain size after DRX decreases gradually with increasing strain level, especially in the initial deformation stage.

Fig. 5 XRD profiles of the precipitates gathered from electrolytic extraction in (a) the as-received sample and (b) the sample after solution treatment at 1473 K for 300 s followed by annealing at 1423 K for 300 s.

Fig. 6 Flow behavior of Co-29Cr-6Mo-0.23C-0.14N alloys in hot compression tests. True stress–true strain curves (a) at a constant deformation temperature of 1423 K at various strain rates and (b) at a constant strain rate of 1 s^{-1} at various temperatures.

Fig. 7 (a) Microstructure of a deformed sample at 1423 K at 1 s^{-1} at a reduction rate of 60%, (b) mean grain size as a function of reduction rate.

Figure 8(a) shows the misorientation angle distribution of the microstructure when deformed at 1423 K, 1.0 s^{-1} at various strain levels. Figure 8(b) shows the fraction of low-angle grain boundaries (LAGBs) and the fraction of $\Sigma 3$ boundaries as a function of the true strain level. An obvious peak of $\Sigma 3$ twinning boundaries can be observed at all strain levels (Fig. 8(a)), whereas the fraction of LAGBs is very low in all cases, indicating uniform recrystallization behavior in these samples under the current condition. Figure 8(b) shows that the fraction of LAGBs decreases gradually with strain and reaches a minimum value at a reduction rate of approximately 20 to 60% followed by a slight increase at a higher strain level. In contrast, the fraction of $\Sigma 3$ boundaries increases to a peak value of approximately 0.49 at a reduction rate of approximately 20 to 60% and subsequently decreases at higher strain. There are two possibilities to explain the result in Fig. 8(b): one is that with increasing strain level, the formed $\Sigma 3$ boundaries are destroyed and transformed into general high-AGBs (HAGBs) continuously; another explanation might be that the formation of $\Sigma 3$ boundaries becomes more difficult. Both of the abovementioned mechanisms are considered to have occurred concurrently because the decreasing grain size inevitably increases the critical resolved shear stress for the twinning formation process,¹⁵⁾ and the slight increase in the LAGB fraction at a high compression rate may be ascribed to the more difficult formation of $\Sigma 3$ boundaries and the larger fraction of SF bands (corresponding to the LAGBs) that remained stable in the matrix. In fact, stable SF bands at higher strain levels have been observed by Li *et al.* in CCM alloy with N addition.¹⁶⁾

3.5 Effect of strain rate on DRX behavior

Figure 9 shows the relationship between DRXed grain size and strain rate after hot compression tests at a deformation

Fig. 8 (a) Misorientation angle distributions of CCMCN alloy with various strain levels and (b) number fraction of LAGBs and $\Sigma 3$ boundaries as a function of true strain levels deformed at 1423 K and strain rate of 0.1 s^{-1} .

Fig. 9 Strain rate dependence of DRXed grain size at a deformation temperature of 1423 K and a reduction rate of up to 60%.

temperature of 1423 K and a reduction rate of up to 60%. The effect of strain rates on the DRXed grain size is remarkable in the strain rate from 10^{-2} to 1 s^{-1} ; higher strain rate is observed to lead to finer DRXed grain size. The most refined grains in CCMCN alloy are observed at a strain rate of up to 1 s^{-1} . In contrast, at higher strain rate, coarser DRXed grain size is observed with increasing strain rate. In other words, the refining behavior of grains at strain rates of 10 and 30 s^{-1} may differ from that at other strain rates. This tendency was observed not only at 1423 K but also at other deformation temperatures. It should be noted that this DRX behavior in current alloy was not observed in either CCM alloy or CCM alloy with N addition. In the current alloy, the addition of both N and C leading to a large number of M_{23}C_6 carbides is considered to play an important role in the DRX process; variation of strain rate possibly leads to the changing of the

Fig. 10 (a) Misorientation angle distributions of CCMCN alloy with various strain rates and (b) number fraction of LAGBs and $\Sigma 3$ boundaries as a function of strain rate deformed at 1423 K with a reduction rate of 60%.

DRX in which these carbides precipitated. However, the role of these carbides in the DRX process remains unclear; further research is required in the future. The distribution of the misorientation angle at various strain rates after hot compressions (compression at 1423 K and 60%) is shown in Fig. 10(a). The fractions of LGBAs and the $\Sigma 3$ boundary at each strain rate are plotted in Fig. 10(b). A minor effect of the strain rate in the entire distribution of the misorientation angle was observed. However, the fraction of the $\Sigma 3$ boundary is observed to be closely related to the grain size; finer grain size leads to a lower fraction of $\Sigma 3$ boundaries.

3.6 Relationship between crystal grain sizes after DRX and Z factor

It is well known that the DRX behavior of alloys is strongly affected by the deformation conditions. In this section, the relationship between the hot compression conditions and the DRX behavior is expressed in terms of the Zener–Hollomon (Z) parameter. In general, the DRX behavior at high temperatures can be expressed in terms of the strain rate, including the Arrhenius-type temperature-dependent term. The Z parameter was calculated using the following equation, which is valid over the entire stress range used in this study:¹⁷⁾

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) = A[\sinh(\alpha\sigma_p)]^n \quad (1)$$

where $\dot{\epsilon}$ is the strain rate; Q , the apparent activation energy of high-temperature deformation; R , the gas constant; T , the deformation temperature; σ_p , the peak stress; n , the stress exponent; and A , a constant. The apparent activation energy Q is obtained using the following equation:

Table 1 Material constants used in Co-29Cr-6Mo, Co-29Cr-6Mo-0.16N, and Co-29Cr-6Mo-0.23C-0.14N alloys for calculating the activation energy.

	n	α/MPa^{-1}	$Q/\text{kJ}\cdot\text{mol}^{-1}$
Co-29Cr-6Mo	4.68	0.0056	562
Co-29Cr-6Mo-0.16N	7.26	0.0026	638
Co-29Cr-6Mo-0.23C-0.14N	6.08	0.0039	678

Fig. 11 Relationship between Z parameter and DRXed grain size.

$$Q = R \cdot \left. \frac{\partial \ln \dot{\epsilon}}{\partial \ln[\sinh(\alpha\sigma_p)]} \right|_T \cdot \left. \frac{\partial \ln[\sinh(\alpha\sigma_p)]}{\partial (1/T)} \right|_{\dot{\epsilon}} = R \cdot n \cdot \left. \frac{\partial \ln[\sinh(\alpha\sigma_p)]}{\partial (1/T)} \right|_{\dot{\epsilon}} \quad (2)$$

where α is the fitting parameter. A good linear relationship is obtained when $\alpha = 0.0039$. Table 1 presents the material constants used to calculate the activation energy for CCMCN alloys. The activation energy for high-temperature deformation is approximately $678 \text{ kJ}\cdot\text{mol}^{-1}$, which is greater than that for the self-diffusion of pure cobalt ($440 \text{ kJ}\cdot\text{mol}^{-1}$) and CCM alloy ($562 \text{ kJ}\cdot\text{mol}^{-1}$) or CCM alloy with N addition ($638 \text{ kJ}\cdot\text{mol}^{-1}$), implying that the interdiffusion of the matrix elements is possibly retarded due to the addition of C and N elements.

The mean grain size (d) after DRX as a function of the Z factor is shown in Fig. 11. At lower strain rate, $\log d$ is roughly linear with $\log Z$. However, at higher strain rate, it is clear that these two parameters cannot be expressed by the same behavior; they are expressed by other linear behaviors with high slope values. It has been reported that the input energy during deformation is partially converted to heat due to adiabatic heating. The latter results in softening and a decrease in flow stress as compared to ideal isothermal deformation conditions.¹⁸⁾ The temperature rise due to adiabatic heating during deformation was also calculated in this research; it was found that the highest temperature rise due to the adiabatic heating of all conditions was approximately 50 K. It is thus considered that other factors, including the precipitates, may play an important role in the DRX process because a temperature rise of approximately 50 K is not very high and is not sufficient to result in grain growth in a high strain rate condition.

4. Conclusions

In this research, the DRX behaviors of CCMCN alloy at temperatures of 1273 to 1473 K and strain rates of 0.01 to 30 s^{-1} were investigated by means of EBSD, SEM, and EPMA. The main results obtained were as follows:

(1) The γ phase in Co-29Cr-6Mo alloy could be stabilized by the slight addition of C and N elements. Numerous precipitates were observed in CMMCN alloy; however, they were identified to be an $\text{M}(\text{Cr}, \text{Mo})_{23}\text{C}_6$ compound and a tiny quantity of σ phase according to EPMA results.

(2) With an increase in strain, a gradual decrease in grain size was observed. The strain rate was observed to have a complex influence on the grain refining process. At lower strain rate, decreasing grain size could be observed with increasing strain rate; however, a coarsening of grain size was observed when the strain rate was higher than 10 s^{-1} .

(3) Higher apparent activation energy Q was observed in CCMCN alloy as compared to that in pure Co, CCM alloy, and CCM alloy with N addition; this was ascribed to the addition of C and N elements, which is considered to retard the self-diffusion of the matrix alloy.

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