

# Effect of Hot Deformation on the Nitride Precipitation Behavior in High Nitrogen Austenitic Steel

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High nitrogen CrMn austenitic steel specimens were compressed at 1000 °C and  $0.1 \text{ s}^{-1}$  to different strains and then held at 850 °C for 360 s. The nitride precipitation behavior after the deformation was investigated using physical simulation, x-ray diffraction, and quantitative metallography. The results showed that the precipitate in 18Mn18Cr0.5N steel after the deformation and holding was  $\text{Cr}_2\text{N}$ . The amount of  $\text{Cr}_2\text{N}$  precipitate was significantly affected by the defect density. Precipitation was promoted by the hardening matrix compared to the recrystallized matrix.

**Keywords** defect density, high nitrogen steel, hot deformation, nitride precipitation, recrystallization

## 1. Introduction

High nitrogen austenitic steel (HNAS) (N levels exceed approximately 0.4 mass%) is developing into a new class of engineering material (Ref 1-4). Recent advances in melting technologies, such as pressurized electric slag remelting and contrary pressure casting manufacture, have made it possible to produce HNAS with N concentrations in excess of 1.0 mass% (Ref 4). As a high nitrogen CrMn austenitic stainless steel, 18Mn18Cr0.5N has an interesting combination of mechanical, chemical, and physical properties: high strength and toughness, good corrosion resistance and low magnetic susceptibility (Ref 5). Thus, it has been widely used for the production of heavy generator retaining rings.

However, surface cracks occur easily during the forging process of 18Mn18Cr0.5N steel retaining rings, leading to high production costs and low efficiency. HNAS is thermodynamically unstable and susceptible to nitride precipitation in the temperature range of 600 to 1050 °C (Ref 6). Secondary phase precipitation generally has significant influence on the hot workability of high alloy steels. Moreover, nitride precipitation significantly reduces material plasticity and results in embrittlement under uniaxial and impact loading (Ref 7). Although Simmons et al. (Ref 7-9) determined the effects of isothermal

aging on  $\text{Cr}_2\text{N}$  precipitation characteristics, the effects of hot deformation degree on the nitride precipitation in HNAS during forging and subsequent holding have not been clarified yet.

In fact, there is a distinct difference in deformation degree between the surface and interior of heavy forgings. In other words, the deformation is unevenly distributed along the section of the forging. Therefore, the present work aims to examine the effect of deformation degree on the nitride precipitation behaviors in 18Mn18Cr0.5N steel during the forging and subsequent holding, and to provide warrants for working out the hot working process of the HANS forgings.

## 2. Experimental Procedures

The test material (18Mn18Cr0.5N steel) was melted in a vacuum induction furnace. After electroslog remelting its chemical composition is as follows (wt.%): 0.09C, 18.7Mn, 18.51Cr, 0.52N, 0.81Si, 0.048V, 0.025P, 0.005S, 0.011Al, and balance Fe. The compression test specimens with a height of 15 mm and a diameter of 8 mm were machined from a forged ingot. Hot compression tests were conducted on a Gleeble3500 thermal/mechanical simulator. The specimens were preheated at 1100 °C for 5 min. They were then cooled to 1000 °C at  $5 \text{ °C s}^{-1}$  and deformed at  $0.1 \text{ s}^{-1}$  to strains of 10, 20, 30, 40, and 70%, respectively. The specimens were cooled to 850 °C and held for 360 s after deformation, then quenched into water immediately.

The deformed samples were cut along compression direction and etched with aqua regia. The microstructures were observed by optical microscopy. The observation site for quantitative metallography was selected, namely, at 1/2 position from the end face to the center of the deformed specimen along the axis, where the deformation conditions were coincident with the setting conditions on the whole (Ref 10). Phase analysis of the specimens was carried out on a Rigaku D/max-2500PC x-ray diffractometer. The tube voltage and current were 40 kV and 40 mA, respectively.  $\text{Cu K}\alpha_1$  ( $\lambda = 0.15406 \text{ nm}$ ) radiation was used. The scanning speed was  $2^\circ \text{ min}^{-1}$ . The volume fraction of nitride precipitate was determined by quantitative metallography method.

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### 3. Results and Analysis

#### 3.1 Precipitate Analysis Results

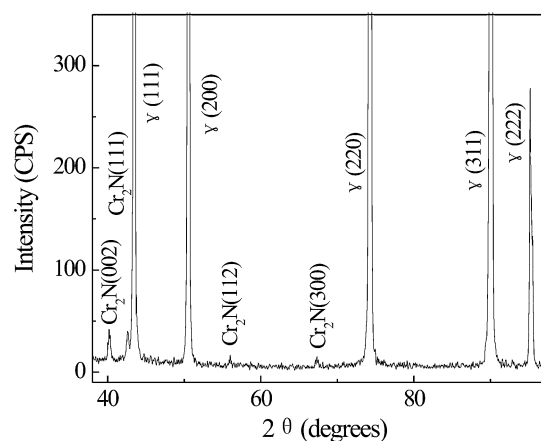
Figure 1 shows the XRD pattern of the 10% deformed specimen held for 360 s at 850 °C. From the results, it is found that the microstructure consists primarily of austenite and less Cr<sub>2</sub>N.

#### 3.2 Microstructures Under Different Deformation Amount Conditions

Figure 2 shows the optical microstructures ( $\times 200$ ) of the quenched specimens held at 850 °C for 360 s with different reductions. It can be seen that the microstructure of the undeformed specimen consists of austenite ( $\approx 90$   $\mu\text{m}$ ) and a few Cr<sub>2</sub>N particles. Moreover, the sample exhibits numerous annealing twins (Fig. 2a). Some austenite grains with serrated grain boundaries are clearly observed in the 10% reduction specimen. In addition, more coarse Cr<sub>2</sub>N particles are dispersed in austenitic matrix (Fig. 2b). With 30% reduction, the austenite is partially recrystallized. Both the amount and size of Cr<sub>2</sub>N particles are smaller than that in Fig. 2(b). Complete recrystallization has occurred in the 40% reduction specimen (Fig. 2c), wherein austenite grain size is about 17  $\mu\text{m}$ . However, the Cr<sub>2</sub>N precipitation is similar to that in Fig. 2(a).

#### 3.3 Nitride Precipitation Behaviors Under Different Deformation Conditions

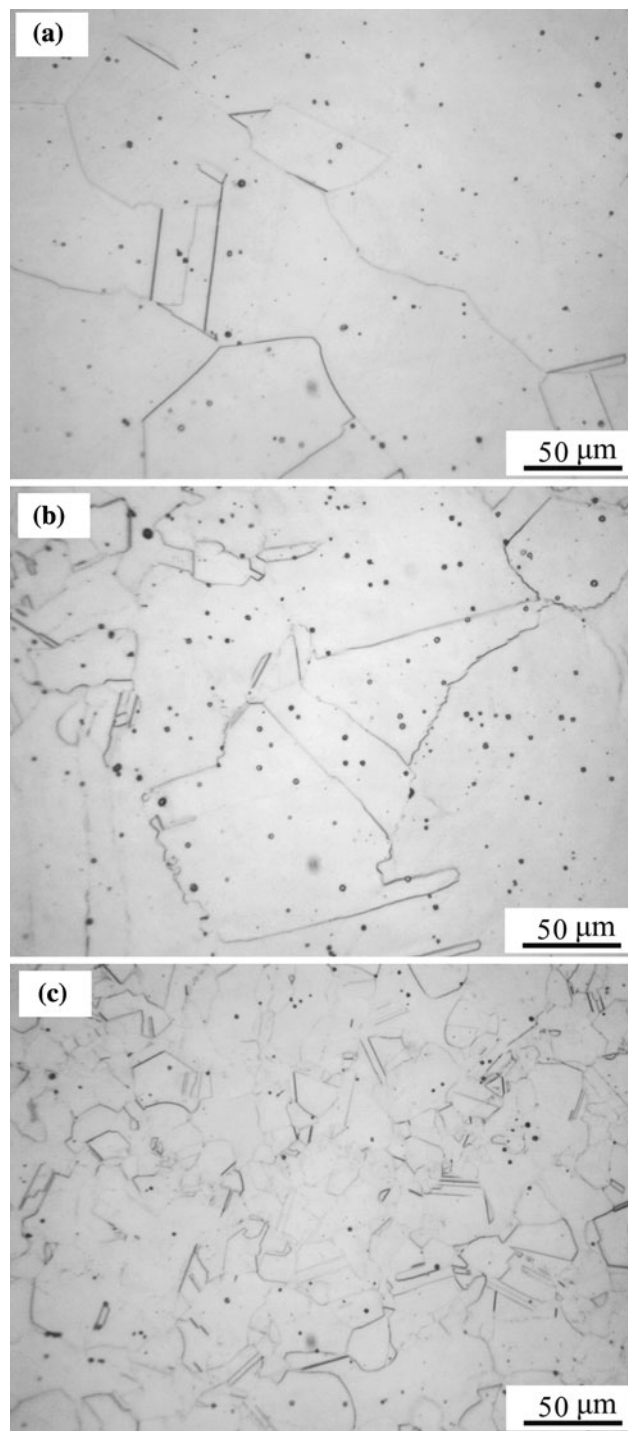
Relationships between the volume fraction of Cr<sub>2</sub>N particles and deformation degree are shown in Fig. 3. It is obvious that the deformation degree at 1000 °C affects the Cr<sub>2</sub>N precipitation kinetics at 850 °C significantly. The volume fraction of nitride particles reaches a maximum (about 1.2 vol.%) in the 10% reduction specimen, and then decreases with increasing deformation degree. Precipitation is promoted by the undeformed matrix compared to the 40 and 70% reduced matrices. Although there may be errors in quantitative metallographic measurements, qualitative relationships between deformation degree at 1000 °C and the amount of Cr<sub>2</sub>N particles at 850 °C can be found.



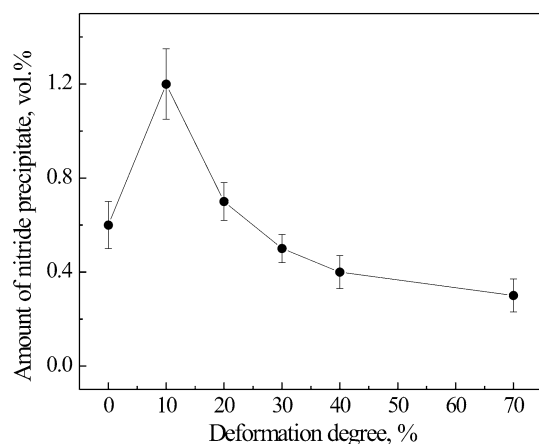
**Fig. 1** XRD pattern of the 10% deformed specimen held at 850 °C for 360 s

### 4. Discussions

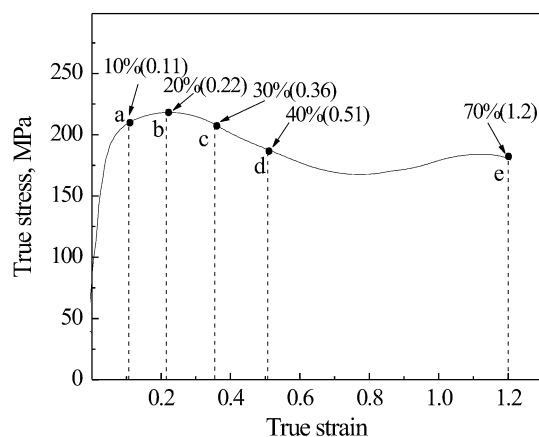
In fact, the decomposition of the nitrogen supersaturated austenitic matrix by precipitation of Cr<sub>2</sub>N particles is a diffusion process of nitride-forming elements. If the diffusion path is different, the Cr<sub>2</sub>N particles nucleation and growth process will also be different. Figure 2 shows that the microstructure features, such as grain size, recrystallization



**Fig. 2** Optical microstructures ( $\times 200$ ) of quenched specimens held at 850 °C for 360 s with different reductions: (a) 0% (undeformed); (b) 10%; and (c) 40%



**Fig. 3** Relationships between the volume fraction of  $\text{Cr}_2\text{N}$  particles and deformation degree



**Fig. 4** Flow curve of 18Mn18Cr0.5N steel determined at 1000 °C and 0.1  $\text{s}^{-1}$

fraction, and defect density etc., vary with the deformation degree. Thus,  $\text{Cr}_2\text{N}$  precipitation characteristics are different in the specimens with various reductions (Fig. 3).

Figure 4 shows the flow curve of 18Mn18Cr0.5N steel determined at 1000 °C and 0.1  $\text{s}^{-1}$ , where points *a*, *b*, *c*, *d*, and *e* denote 10, 20, 30, 40, and 70% deformation, respectively. Numbers in brackets are the corresponding true strains. Point *b* is also the peak strain ( $\epsilon_p$ ). Normally, the expression to estimate the relationship between the critical strain  $\epsilon_c$  at which for the austenite to start dynamic recrystallization (DRX) and the peak strain is defined as  $\epsilon_c = 0.8\epsilon_p$  (Ref 11). The critical strain for DRX for 18Mn18Cr0.5N steel, at 1000 °C and 0.1  $\text{s}^{-1}$ , is thus between 10 and 20%.

With 10% reduction (point *a*), the deformation induced defects (such as vacancy and dislocation etc.) are not abundant enough to induce DRX. Softening by static recovery is very small after deformation and subsequent holding (Ref 12). In addition, static recrystallization has not taken place, whose incubation period is more than 1.0 ks under this condition (Ref 13). The defect density is evidently high during the holding process. Defects are favorable for the diffusion of nitride-forming elements. Therefore, the  $\text{Cr}_2\text{N}$  precipitation occurs easily in 10% reduction specimen. After 20% reduction (point *b*), the strain is high enough to lead to DRX.

Some defects thus annihilate due to DRX and metadynamic recrystallization. Moreover, static recrystallization also occurs to some extent. The defects are annihilated in large numbers through the combination effects of DRX, metadynamic recrystallization, and static recrystallization. The diffusion paths are reduced significantly, and consequently  $\text{Cr}_2\text{N}$  precipitation is restrained. The stress decreases markedly up to 30% or even 40% reduction (points *c* and *d*), because the rate of defect annihilation is higher than generation. After 70% reduction (point *e*), full DRX has occurred, and renewed hardening and softening have also taken place. In the 40 and 70% reduction specimens, plenty of defects are annihilated by DRX and metadynamic recrystallization, and a few by static recrystallization, leading to less  $\text{Cr}_2\text{N}$  precipitation. It can be concluded that  $\text{Cr}_2\text{N}$  precipitation is significantly affected by defect density. The higher the density is, the more easily the  $\text{Cr}_2\text{N}$  precipitation may occur.

It is suggested that the minimum extent of deformation is about 30% to restrain nitride precipitation in the hot working of 18Mn18Cr0.5N steel. The effect of nitride precipitation on the surface cracking and the hot workability of HNAS will be considered in the further work.

## 5. Conclusions

The precipitate in 18Mn18Cr0.5N steel after the deformation and holding was  $\text{Cr}_2\text{N}$ . The amount of  $\text{Cr}_2\text{N}$  precipitate was significantly affected by the defect density in the matrix. Precipitation was promoted by the hardening matrix compared to the recrystallized matrix.

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