



Mechanism for the formation of Z-phase in 25Cr-20Ni-Nb-N austenitic stainless steel



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ABSTRACT

Precipitation behavior of Z-phase (NbCrN) is investigated in 25Cr-20Ni-Nb-N austenitic stainless steel during long-term aging. The early precipitation of secondary NbN appears when the steel is aged for 100 h, and the secondary NbCrN precipitates adhere to the pre-existing NbN particles during the subsequent aging. A semi-coherent relation between $(1\ 1\ 1)_{\text{NbN}}$ and $(1\ 1\ 2)_{\text{NbCrN}}$ is identified, which provides favorable sites for the nucleation of secondary NbCrN on secondary NbN. Subsequently, secondary NbN will dissolve to supply niobium and nitrogen for the growth of secondary NbCrN.

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1. Introduction

The resistance to high operating temperature and steam pressure allows 25Cr-20Ni-Nb-N austenitic stainless steel to be widely used in superheaters and reheaters, increasing energy efficiency and reducing pollution for ultra-super critical (USC) power plants [1,2]. This austenitic stainless steel exhibits excellent creep properties, owing to the precipitation strengthening from finely dispersed Z-phase at high temperature [3].

The Z-phase, a complex nitride (NbCrN), was first observed in austenitic steels in the 1950 s [4]; Primary NbCrN with micrometer size precipitated in casting during solidification, and the intragranular particles with nanometer size were identified to be secondary NbCrN in aged S31042 steel at 700 °C [5]. The secondary NbCrN is fine and relatively stable during long-term service [6]. Zhang et al. [7] found that worm-shaped Z-phase entangled with dislocations was the main contributor to the precipitation strengthening.

On the other hand, the precipitation of Z-phase caused a detrimental effect on the creep strength of martensitic steel [10], thus many papers have reported studies of the nucleation of Z-phase in 9–12% Cr steels. Compared with the original Z-phase in austenitic steel, the tetragonal unit cell with a smaller lattice parameter ($a = 0.286$ nm and $c = 0.739$ nm) is regarded as modified Z-phase in martensitic steels, due to the replacement of niobium by vanadium [8,9]. Two different formation mechanisms of Z-phase

have been proposed, i.e. nucleation and growth mechanism [11,12], and direct transformation of MX into Z-phase by Cr diffusion from matrix into MX [13,14].

Few studies have addressed the precipitation behavior for Z-phase in austenitic stainless steel so far, though the existence of Z-phase has been confirmed [6,7]. The present work focuses on the crystal structure and nucleation site of Z-phase in 25Cr-20Ni-Nb-N austenitic stainless steel during high-temperature exposure, especially the crystallographic orientation relationship between MX and Z-phase. Detailed mechanism for the formation of Z-phase is proposed.

2. Experimental

The material for this study is 25Cr-20Ni-Nb-N austenitic stainless steel with the following composition in wt.%: C 0.07, Mn 1.16, Si 0.38, Cr 24.94, Ni 20.49, Nb 0.44, N 0.26, Cu 0.05, and Fe in balance. The specimens were solution treated at 1250 °C for 30 min, followed by water quenching. The aging treatment was then carried out at 700 °C for a different time spans of 100 h, 500 h, 1000 h, and 3000 h. The metallographic specimens were mounted in resin, grounded by SiC paper and polished by flannelette, then etched with a solution of HCl (20 ml), Ethanol (20 ml) and CuCl₂ (5 g) for less than 1 min. The microstructure of solution treated and as-aged samples was observed by means of Hitachi S4800 scanning electron microscope (SEM). The precipitates, extracted by chemical method using a mixed solution of 50% hydrochloric

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acid and 50% deionized water, were identified by a Bruker D8 Advanced X-ray diffraction (XRD). To observe and confirm the precipitates, thin foils were mechanically thinned down to 50 μm , and followed by double-jet electrolytic polishing in a solution of 5% perchloric acid and 95% ethanol at $-30^\circ\text{C}/30\text{V}$. The foils were examined by a JEM 2100f transmission electron microscope (TEM) operating at 200 kV.

3. Results

Fig. 1a shows the XRD patterns of the extracted powder of the solution treated sample and the samples aged at 700°C for up to 3000 h. The main precipitates are Z-phase and MX. The Z-phase with a NbCrN composition has a tetragonal crystal structure, with the lattice parameters of $a = 0.428\text{ nm}$ and $c = 0.736\text{ nm}$, and the actual composition of the MX is face-centered cubic (fcc) NbN, with $a = 0.439\text{ nm}$. The precipitates in the solution treated sample are primary NbCrN and NbN, which cannot be dissolved at the current solution temperature. Therefore, the aged samples contain secondary NbCrN and NbN phases, as well as the primary ones,

considering the significantly increased peak intensities of NbCrN and NbN. The summary of quantitative analysis of the XRD patterns is indicated in Fig. 1b. At the initial stage of aging (0–100 h), the relative content of NbN increases as that of NbCrN decreases, which results from the secondary-precipitated NbN. When the aging is over 100 h, the relative content of NbN and NbCrN, increases and decreases in turn, respectively, indicating that NbCrN is formed from NbN during the long-term aging.

The SEM micrographs of 25Cr-20Ni-Nb-N steel before and after aging are shown in Fig. 2. The solution-treated alloy is composed of an austenitic matrix and micron-sized primary nitride phases, in which the precipitates with irregular shape should be the undissolved NbCrN, and the minor particles are NbN (Fig. 2a). Some primary NbCrN distributes discontinuously along grain boundaries due to its higher precipitation temperature than the critical point, austenitic grain boundaries are incompletely wetted by primary NbCrN [15,16]. An increasing number of rectangular NbN, with the size of 20–50 nm, precipitated inside the grains of the sample aged for 100 h (Fig. 2b). As the aging time increases to 1000 h, rod-like precipitates was newly formed at the sites of NbN, and one could identify a certain orientation between them (Fig. 2c).

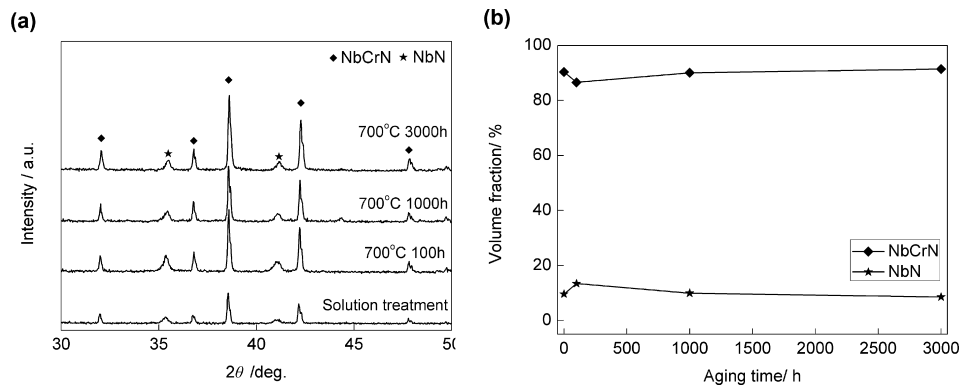


Fig. 1. (a) X-ray patterns and (b) changes in volume fraction of the extracted precipitates from the solution treated and as-aged samples.

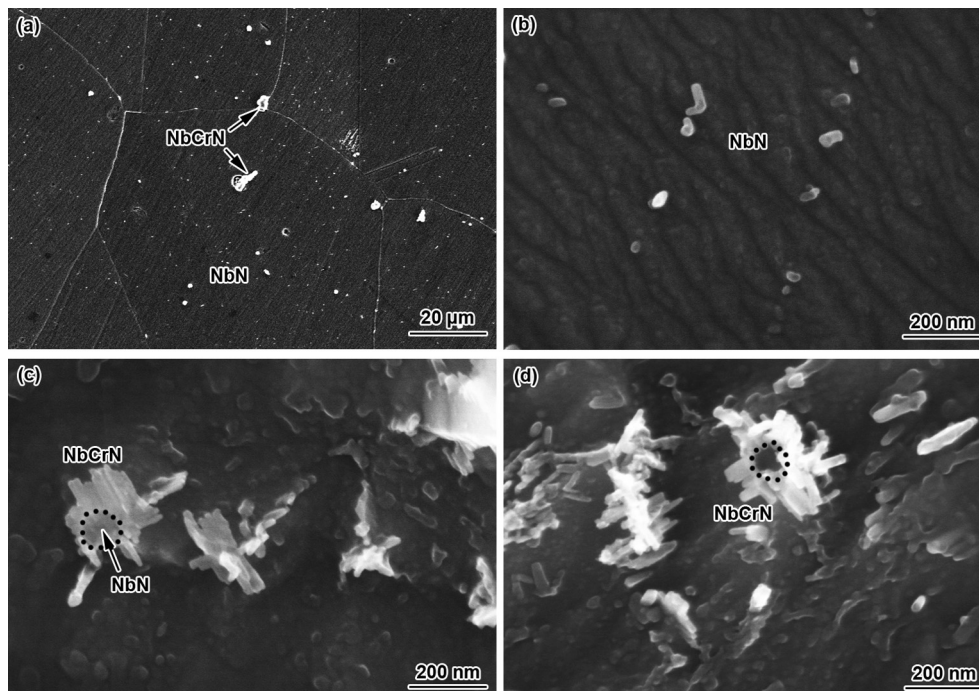


Fig. 2. SEM micrographs of (a) the solution treated sample and the samples aged at 700°C for (b) 100 h, (c) 1000 h and (d) 3000 h.

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