Research on Microstructure Transformation of Super304H Stainless Steel in the Process of Aging at 700℃

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Abstract—To study microstructure transformation of Super304H, the original sample and the samples aging for 500h, 800h, 1500h, 2500h and 3650h at 700℃ were analyzed with OM, SEM, TEM and X-ray diffractometry. The results showed the grain size and the precipitated phases along boundaries increased as aging time prolonged, the area fraction and the amount of precipitated phases increased remarkably, while the type of precipitated phases changed from $M_{23}C_6$ to $Cr_{23}C_6$ and Nb (C,N) as aging time varied from 500h to 3650h. The functional relation between area fraction of precipitated phases and aging time at 700℃ was also given.

***Keywords—Super 304H; Microstructure; Transformation; Aging;* Precipitated Phase**

I. INTRODUCTION

Super304H stainless steel has been widely used in hypercritical and ultra-supercritical power units for its comprehensive performance at high temperature [1-3]. But long time working at high temperature and high pressure will lead to its microstructure transformation and aging which could cause facility failure [4-5]. Compared to TP304H, TP347HFG and other heat resistant austenitic stainless steels, some elements, such as Cu, Nb and N in Super304H, make the formation of precipitation of carbide and intermetallic compounds much more complicated [4-11]. It's significant to research the microstructure transformation of Super304H austenitic stainless steel for supervising the damage level of tubes in service.

II. EXPERIMENTAL METHOD

Original Super304H stainless steel tube used for this aging experiment is in accordance with the standard of ASTM A-213M (TABLE I). Its specification is OD45mm ×8mm. Using wire-electrode cutting machine, six tube-sections with 200mm long were obtained, and five of them were aging treated for 500h, 800h, 1500h, 2500h and 3650h respectively. To accelerate the microstructure transformation, the aging temperature was 700℃. Then six samples were analyzed with OM, SEM, TEM and X-ray diffractometry successively. The instrument as showed in Fig.1 was designed and used to extract precipitated phases powder for X-ray diffraction analysis.

TABLE I. Composition of Super304H

Composition (wt%)	Super304H (ASTM A-213M)	Actual value
C	0.07～0.13	0.10
Si	≤0.03	0.25
Mn	≤1.00	0.80
P	≤0.040	0.032
S	≤0.010	0.032
Cr	17.00～19.00	18.3
Ni	7.50～10.50	8.62
Mo	-	0.16
Cu	2.50～3.50	2.38
Nb	0.20～0.60	0.49
N	0.05～0.12	-

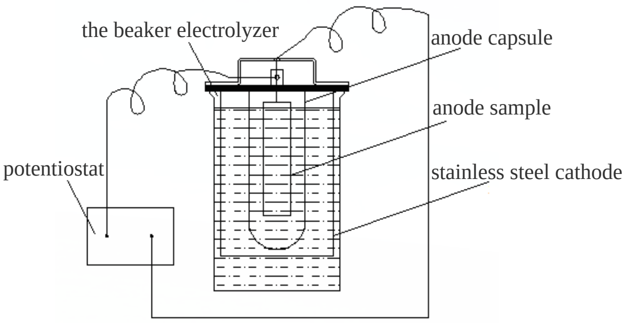
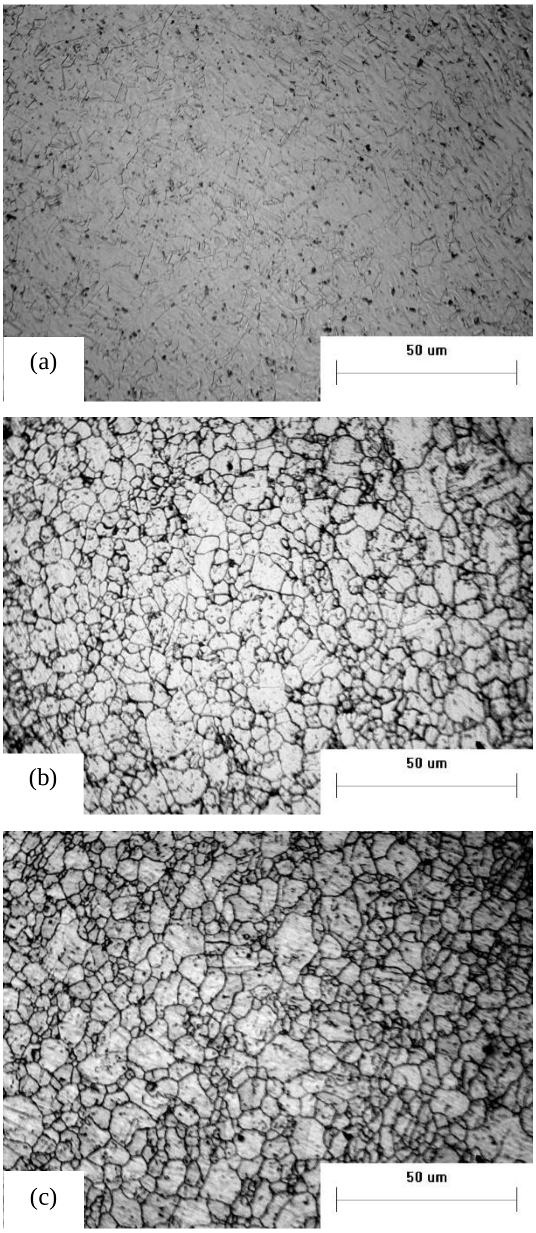


Figure 1. Instrument for Precipitated Phases Powder Extraction

III. DISCUSSION



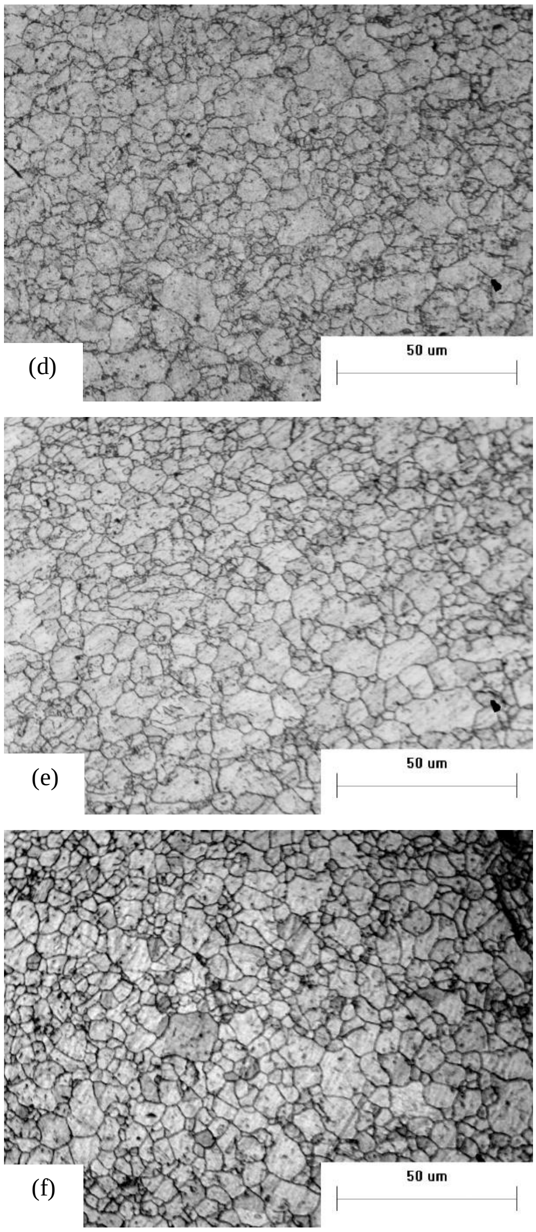
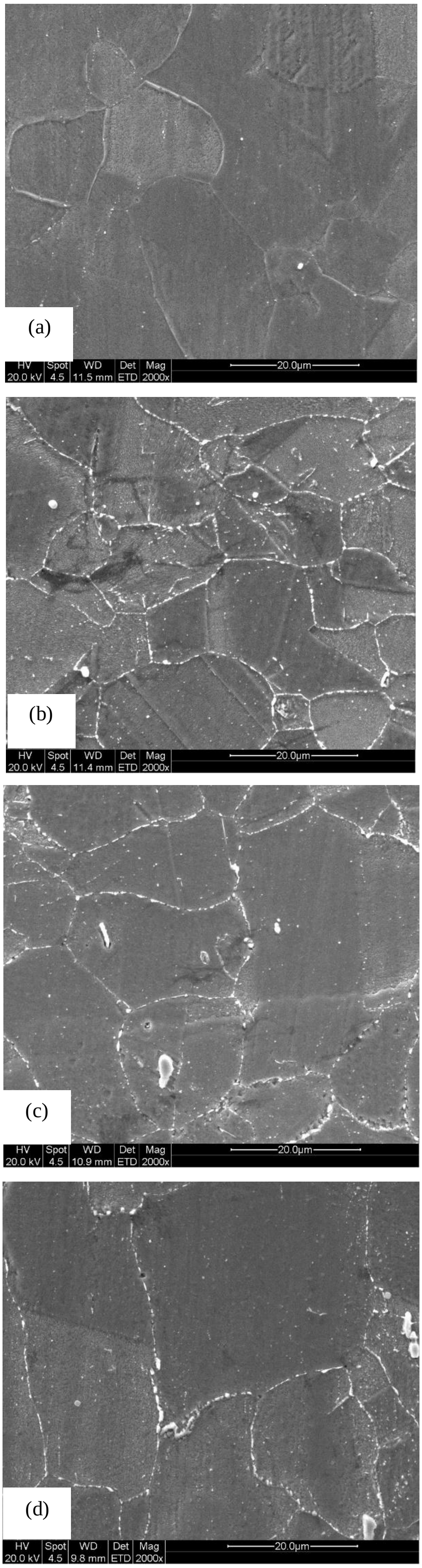


Figure 2. Metallurgical Structure of Original and Aging Treated Super304H Samples
(a)Original, (b)Aging for 500h, (c)Aging for 800h, (d)Aging for 1500h, (e)Aging for 2500h, (f) Aging for 3650h

Fig.2 (a) ～ (f) show the optical microstructure (especially twins) transformation of Super304H aging for different time at 700℃. The original sample has fine and multitudinous twins, and their boundaries are linear. After being treated for 500h, the size of twins increased, while their number decreased. After aging for 800h, the grains grew up consistently, their size tended to be identical, and those with abnormal dimension increased in number as well. Then the twins almost diminished, while large grains emerged and precipitated phases distributed along boundaries after 3650h aging treatment.



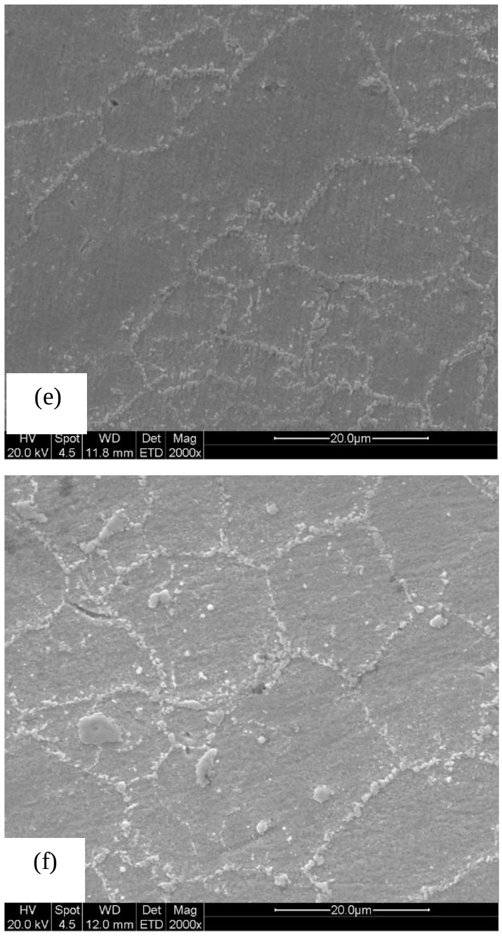


Figure 3. Microstructure of Original and Aging Treated Super304H Samples with SEM
(a)Original, (b)Aging for 500h, (c)Aging for 800h, (d)Aging for 1500h, (e)Aging for 2500h, (f) Aging for 3650h.

It is revealed from Fig.3(a)～(f) that the precipitated phases along boundaries increased dramatically as aging time prolonged. Compared with Fig. 2, we can see that the patterns under OM and SEM are corresponding. Based on quantitative analysis of those photographs, the curves of relationship between the amount, the average size and the area fraction of precipitated phases with aging time could be given as Fig.4 and Fig.5.

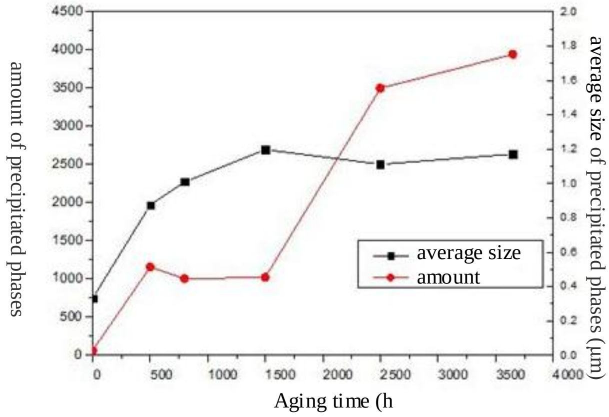


Figure 4. The Curve of Relationship between the Amount and the Average Size of Precipitated Phases with Aging Time

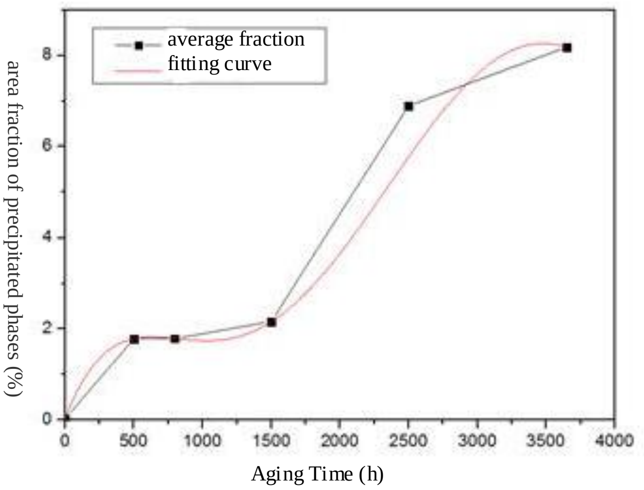
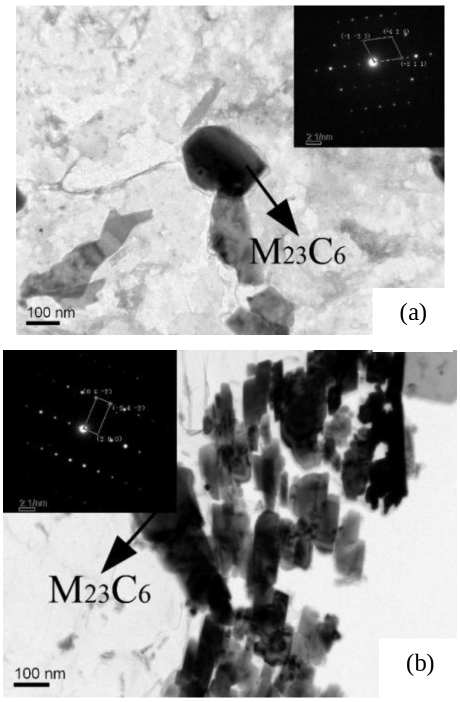


Figure 5. The Curve of Relationship between the Area Fraction of Precipitated Phases with Aging Time

The functional relation between the area fraction of precipitated phases (y, unit: %) and aging time (x, unit: h) can also be derived from the fitting curve in Fig.5. That is

$$y = 0.036 + 0.78\times10^{-3}x - 1.20\times10^{-5}x^2 + 7.61\times10^{-9}x^3 - 1.85\times10^{-12}x^4 + 1.53\times10^{-16}x^5 \quad (1)$$

From Fig.4 and Fig.5, we can see the amount and average size of the precipitated phases increased as aging time prolonged. The highest incremental speed of the amount emerged when the sample treated for 1500h to 2500h. Meanwhile, after aging for 1500h or longer, the average size of precipitated phases became stable.



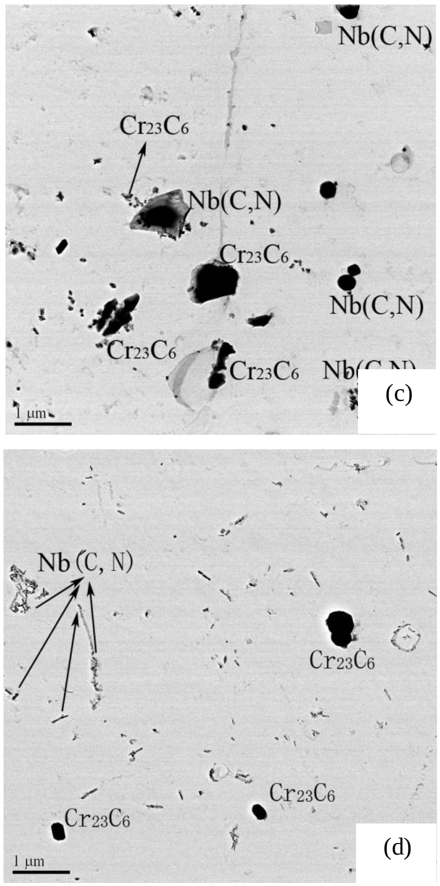


Figure 6. Patterns and Calibration of the Precipitated Phases in the Samples with Different Aging Time at 700℃
(a) Aging for 500h, (b)Aging for 1500h, (c)Aging for 2500h, (d) Aging for 3650h

Furthermore, we analyzed TEM patterns and calibrated the diffraction patterns of precipitated phases in the samples with different aging time at 700℃ (Fig.6). The results showed that the main precipitated phases in the sample being treated for 500h are scattered and fine M23C6, while those in the sample treated for 1500h are clustered M23C6. When the sample being treated for 2500h and 3650h, the precipitated phases became clustered Cr23C6, fine and scattered Nb (C, N).

IV. CONCLUSION

(1) As aging time prolonged at 700℃, the grain size of Super304H increased and the precipitated phases along boundaries manifolded.

(2) In the samples being treated for 1500h and 2500h at 700℃, the area fraction and the amount of precipitated phases increased remarkably.

(3) The type of precipitated phases changed from M23C6 to Cr23C6 and Nb (C, N) as aging time varied from 500h to 3650h at 700℃.

(4) The functional relation of area fraction of precipitated phases (y, unit: %) with aging time (x, unit: h) at 700℃ could be given as

$$y = 0.036 + 0.78\times10^{-3}x - 1.20\times10^{-5}x^2 + 7.61\times10^{-9}x^3 - 1.85\times10^{-12}x^4 + 1.53\times10^{-16}x^5.$$

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