Mansoura Engineering Journal

Volume 19 | Issue 4 Article 20

12-1-2021

Morphology of Discontinuous Precipitates in Nitrogenized 316 L Steel.

Fawzy El-Sabbahy
Assistant Professor of Metallurgical Engineering Department., Faculty of Petroleum and Mining Engineering., Suez., Egypt.

Mansoura Engineering Journal (MEJ), Vol. 19, No. 4, Dec. 1994 M 80

MORPHOLOGY OF DISCONTINUOUS PRECIPITATES IN NITROGENIZED 316 L STEEL

شكل وتوزيع الترسيبات الغير متصلة فى صلب ٣١٦ـل النتروجينى

F.N. El-Sabbahy
Department of Metallurgical Engineering, Faculty of Petroleum and Mining Engineering, Suez, Egypt.

خلاصة عربى :

عينات مسحوق صلب ٣١٦ـل المحتوية على ١٨٪ كروم - ٨٪ نيكل - ٢٪ موليبدنم وكذا النتروجين فى النطاق من ٠,٠٢٪ إلى ٠,٦٪ تمت معالجتها بالتسخين عند درجة حرارة ١١٠٠ °م لمدة ٣٠ دقيقة ثم طشها مباشرة فى الماء. العينات المعالجة حراريا تم تعتيقها عند درجات حرارة ٧٠٠ °، ٨٠٠ °م لمدد مختلفة من الوقت. ولدراسة شكل وتوزيع الترسيبات تم استخدام الميكروسكوب الالكترونى المساح والأشعة السينية. وكذلك تم قياس الصلاده فى مختلف حالات التعتيق. ولقد وجد أن شكل وتوزيع وحجم الترسيبات يعتمد على متغيرات التعتيق (درجة الحرارة والوقت) وأيضا على محتوى النتروجين.

المدخـــلات : صلب ٣١٦ـل - معالجة الإذابة - التعتيق - شكل وتوزيع الترسيبات.

ABSTRACT

316 L steel specimens, wt %, (18 Cr - 8 Ni - 2 Mo) containing nitrogen in the range of 0.02 - 0.6 wt% in powder form, were solution treated at 1100°C for 30 min. followed by water quenching. Solution treated materials were subjected to aging at temperatures of 700 and 800°C for several intervals of time. X-ray diffraction and Scanning electron microscopy were used to study the morphology of the precipitates at different aging conditions. The hardness of these materials was determined. The morphology and size of the precipitates produced during aging were observed to depend on the aging parameters (time and temperature) and nitrogen content.

KEY WORDS : 316 L steel - Solution treatment - aging - Morphology of precipitates.

INTRODUCTION

Nitrogen is often present to some extent in stainless steel as an impurity. However it is used in more significant amounts as an alloying addition. In this case nitrogen has been used as a more economical partial or complete replacement for nickel to promote the presence of austenitic phases in a solid solution lattice as a strengthening element and/or to give increased rate of work hardening of austenitic structure [1].

In ingot metallurgy the strengthening of stainless steel by nitrogen has been discussed in several papers concerning both solid solution and precipitation strengthening. [2-5]. ***Okamot et al.*** [5] studied the variation of hardness and microstructures by heat treatment and rolling. It has been observed that the hardness of solution treated alloys at 1200°C for 1h and that of the as rolled alloy was noticeably increased with the increase of nitrogen concentration in the range of 0.01 to 0.5%. Also, the effect of aging times at various aging temperatures between 600 - 800°C for steel containing 0.02% N on the hardness was investigated [2]. The results showed that the hardness was slightly affected by aging times (up to 100 hrs) at mentioned temperatures. There is no attention given to the precipitate morphology, size and type which may be responsible for the strength variation during the aging processes. Therefore, the aim of this work is to study the effect of nitrogen content, solution treatment and aging on the morphology of the precipitates. Nitrogenized austenitic stainless steel powder was used in the present work.

Experimental work :

The tested material was nitrogenated austenitic stainless steel powder containing 0.02 - 0.6% N produced by plasma technique which was described in details elsewhere [6]. A solution heat treatment was carried out at 1000 and 1100°C for 30 min. followed by water quenching. The solution treated materials were aged at 700 and 800°C for several intervals of time followed by water quenching.

Several techniques have been used to study the aging process of the investigated material. X-ray diffraction Cu-kα, has been used to determine the lattice parameter of austenite matrix at different levels of nitrogen in as solution treated and for aged material at 800°C for different times. Microstructure has been observed using scaning electron microscopy [SEM TA 33] equipped with EDX. Vicker's hardness measurements were used to follow the solution and aging process of the investigated material using 1 kp load. Each value was taken as an a verage of 5 readings.

Results and Discussion:

Solution treatment.

Figure 1 shows the microstructure and morphology of the precipitates in as received powder containing 0.6 % N. The precipitates are observed to be located mainly at the grain boundaries. X-ray diffeaction analysis proved that the preapitates are mainly nitrides [7].

Figure 2 shows the microstructure of solution treated materials at 1050 and 1100°C for 30 min. It is clear that complete solubility of the precipitates is achieved at 1100°C, Fig. 2b. Therefore, the solution treatment at 1100°C was applied for the rest of materials.

The varation of the austenite matrix lattice parameter with the nitrogen content was determined using X-ray diffraction. The results obtained are given in Fig. 3. It is observed that a linear relationship exists betwen the lattice parameter and nitrogen content. This relation can be described by the following equation.

$$a = 0.025\ N\ \% + 3.59.$$

The expansion of the austenite lattice parameter with increasing the nitrogen content is attributed to the presence of nitrogen atom in interstitial sites in FCC austenite lattice.

The hardness of these materials, under the same conditions, was measured. The results obtained are depecited in Fig. 4 was so remarkably increased with the N % in steel , that the Hv about 198 with 0.02% N was raised to Hv 235 with 0.6% N. Such increase in hardness is due to solid solution hardening. The relation between hardness and nitrogen content can be described by the following linear equation.

$$Hv = 60.68\ N\ \% + 196.94.$$

Namely, with increase of 0.1 % N, the hardness was enhanced by about 6 Hv and the lattic parameter was increased by 2.5×10^{-3} A°.

Aging treatment.

Two nitrogen levels, the one containing 0.4% N, and the other 0.6%N were subjected to aging at 700 and 800°C for different times 1,10,50 and 100 hrs.

Fig. 5 shows scanning electron micrographs for steel containing 0.4% N after aging at 700 and 800°C for 1 h. It can be observed that at 700°C the discontinuous precipitation nucleates at the grain boundary and growth outwards as nodules into the matrix phase. Figure 6 shows the discontinuous precipitates after aging at 800°C for 10 hrs. The volume fraction of the precipitates increased with increasing the aging temperature as shown in Fig. 5 b or the aging time, Fig. 6. The aging time is observed to greatly influence the precipitates morphology. Several forms of precipitates can be recognized very iong needles, short needles and fine precipitates. According to Geisler [8], the driving force for growth of discontinuous precipitation stems probably from th straining of the lattice. This straining is caused by thermal effects imposed during the solution treatment and the general precipitation.

Fig. 7 Shows micrographs of steel containing 0.4% and 0.6% N aged at 800°C for 100 hrs. It can be observed that the precipitates take a spherical form instead of needle from obtained at aging times 1 and 10 hrs. Also, the quantitiy of precipitates increases with increasing nitrogen content as can be seen from fig. 7. In Fig. 7a. the precipitates are distributed mainly along the grain boundaries. For higher nitrogen content precipitates are distributed not only along the grain boundaries but also randomly within the grain, Fig. 7b. It is known that the stability of the precipitates is strongly dependent upon their geometrical shape. The spherical form is the most stable one [9]. Therefore with increasing the aging time the discontinuous precipitates changed from the initial less stable form, needle-like to the more stable one, spherical. Discontinuous precipitation can be regarded as a stable form of dispersion hardening as such show a considerable resistance to over aging [10]. This effect can be useful in maintaining high strength levels after high temperature aging or during elevated temperature services.

Hardness measurement results are given in table [1] . There is an initial drop in the hardness value which followed by an increase with the aging time. The initial drop in hardness can be attributed to reversion process which is expected to take place in solution treated materials [11] stored for some times at room temperature before aging at higher temperature. A slight increase in hardness is observed after long aging times at 700 and 800°C for both steels, which is in agreement with previous work [2]. Also after long aging time at the given temperatures it seems that the nitrogen content does not affect the hardness value appreciably.

The effect of aging time at 800°C on the lattice parameter was examined for steel containing 0.6 % N. The results obtained are depicrted in table [2]. It is clear that aging up to 100 hrs leads to a decrease in lattice parameter of austenite matrix. This means that with increasing aging time at 800°C the volume fraction of the precipitates increases. Such results is in a good agreement with previous work [12].

Conclusion :

On the basis of the obtained results one can be concluded that the morphology of precipitates depend on aging parameters (time and temperature). At short aging times and low aging temperatures a needle form precipitates are observed which changed into spherical form at prolonged aging time and/or high aging temperatures. That form is observed to maintain high strength level after high temperature aging and/or prolonged aging time.

Acknowledgement

The author would like to thank **Prof. Badr I. Yostos,** Faculty of Pet. & Mining. Suez Canal University, for the valuable discussions and guidance during this investigation. Also thanks to the Institut für Eisenhüttenkund and the Institut für Werkstofkunde in Aachen- Germany for providing the research facilities.

REFERENCES

1- K.K. Mallick, M.Sc thesis, University of Newcastle upon Tyne (1979).

2- K.J. Irvine; D.T. liewellyn; A.I.M. and F.B. Pickering, "J.Iron and steil Inst.", 10 (1961) P. 153 - 175.

3- H. Thier; A. Baumel and E. Schmidtmann. "Arch. Eisenhüttenwessen", 40 (1969) P. 333 - 339.

4- W. Schumacher, " Conference : New Development in Stainless Steel Technology" Detroit, Micigan, U.S.A., 17 - 21 Sep. (1984) P. 107 - 116.

5- M. Okamoto; R.Tanak; T. Naito and R. Tujimota. "Testu to Hagane Overseas", 2 (1962) P. 25 - 37.

6- F.N.El-Sabbahy, " Ph. D. Thesis" Suez Canal University. Egypt, 1991.

7- E.Wōsh " Diploma Thesis " Rheinish - Westfälische Technische Hochschule, Aachen, Germany, (1986).

8- A. H. Geisler : " Phase Transformation in Solids" (1951), J. Wiley and Sons.

9- J. Burke " The kinetics of Phase Transformation in Metals " Pergamon Press, London, (1965). P. 134.

10- J. W. Martin " Precipitation hardening" Pergamon Press.; London, (1968), P. 112.

11- K.J. Irvine; D.T. liewellyn and F.B. Pickering, "The Metallurgical Evolution of Stainless Steels" Ed. by F.B. Pickering. London, (1987) P. 377.

12- M. Kajihara; Si - Kyung Choi; R. Tanaka; Y. Seo; T. Okumura and Y. Kondoh, " Z. metallkde." 77 (1986). P. 515 - 518.

Table [1]: The effect of aging time and temperature on the hardness of samples solution treated at 1100°C for 30 min.

N (wt %)	Aging temp. (C)	Aging time (h)	Hardness (Hv_1)
0.4	700	as solution treated	220
		1	210
		10	209
		50	217
		100	257
	800	as solution treated	220
		1	200
		10	229
		50	235
		100	260
0.60	700	as solution treated	234
		1	229
		10	229
		50	250
		100	262
	800	as solution treated	234
		1	204
		10	221
		50	229
		100	265

Table [2]: A comparison of the lattice parameter results obtained in the present work and the previous work.

No	N (wt %)	Aging temp. °C.	Aging time hrs	Lattice parameter A°	Reference
1	0.69	800	0.003	3.6060	Kajihara and Co-workers[12]
			3	3.5990	
3	0.6	800	1	3.607	This work
			10	3.605	
			50	3.601	
			100	3.597	

Fig. (1): Scanning electron micrograph for austenitic stainless steel containing 0.6% N melted under plasma. 3000 X

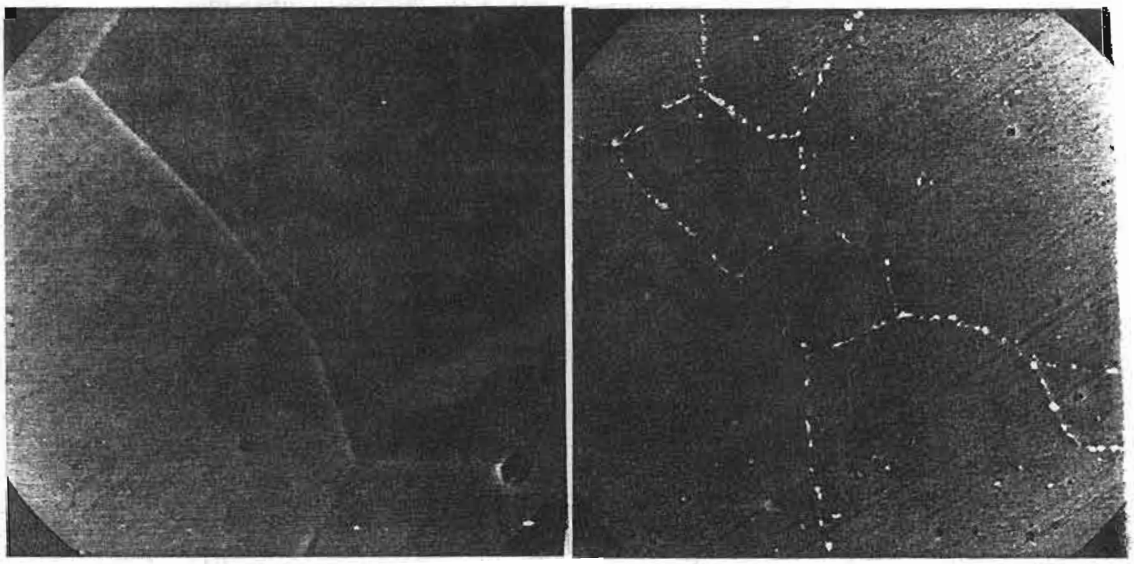

Fig. (2): Effect of solution annealing temperature on the precipitates dissolution in nitrogenized steel containing 0.6% N.
a) 1000°C 2000 X b) 1100°C 3000 X

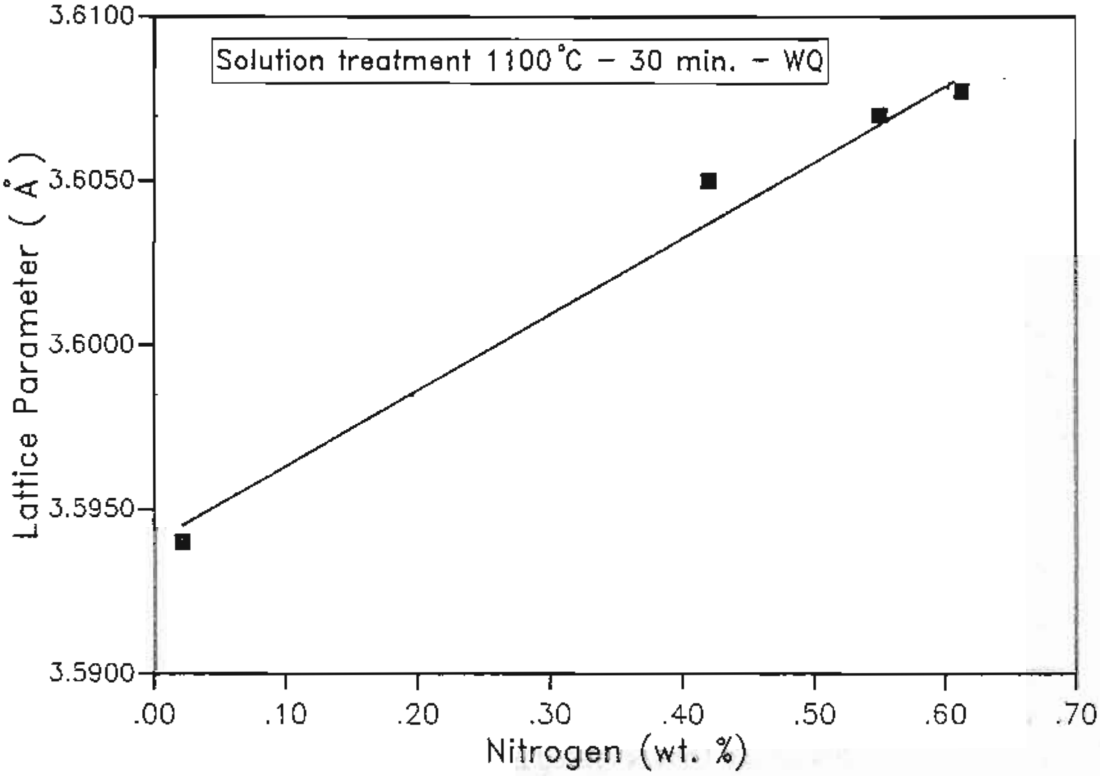

Fig. (3): Effect of nitrogen content on the lattice parameter of austenitic stainless steel.

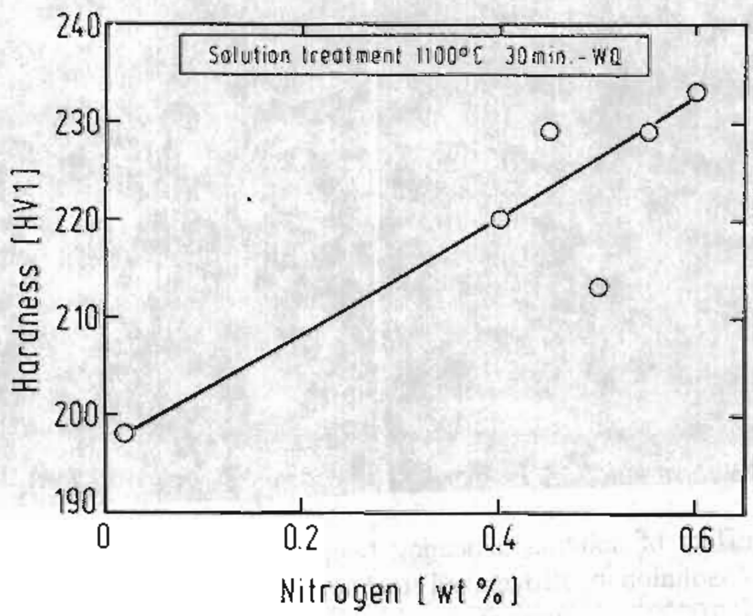

Fig. (4): Effect of nitrogen content on the hardness of solution heat treated samples.

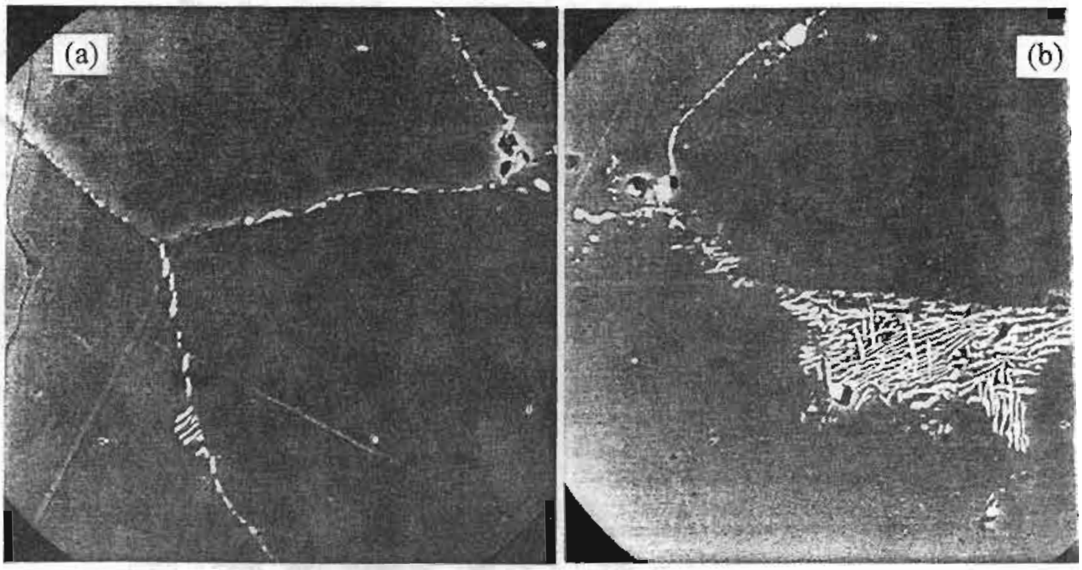

Fig. (5): Discontinuous precipitates in nitrogenized steel containing 0.4%N, solution treated at 1100°C for 30 min. and aged for one hour at
(a) 700°C b) 800°C. 3000 X

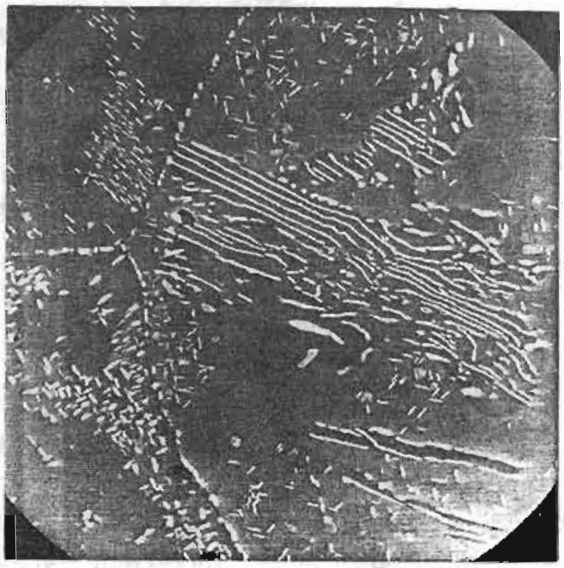

Fig. (6): Intensive discontinuous precipitates in nitrogenized steel containing 0.4% N, solution treated at 1100°C for 30 min. and aged at 800°C for 10 hrs. 3000 X

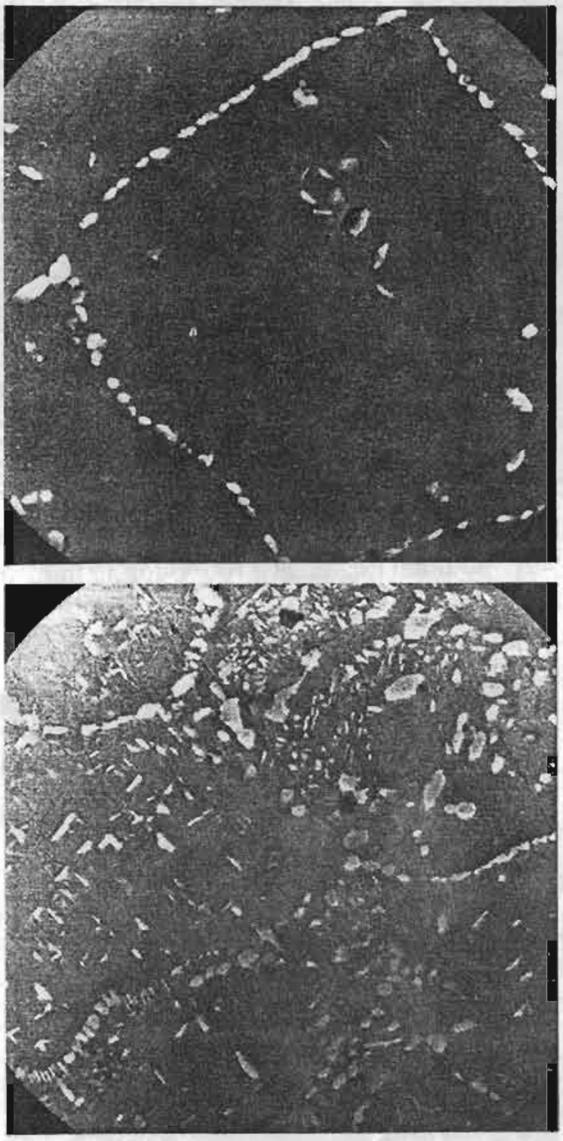

Fig. (7) : Morphology of discontinuous precipitates of austenitic stainless steel solution treated at 1100°C for 30 min. and aged at 800°C for 100 hrs. (a) N = 0.4% (b) N = 0.6%. 3000 X