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THE EFFECT OF NITROGEN ON LOW-CYCLE FATIGUE MECHANISMS OF AUSTENITIC STAINLESS CAST STEELS

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The effect of nitrogen on mechanical properties and low cycle behaviour of Cr-Ni-Mn-Mo-Nb-N austenitic stainless cast-steels has been investigated at room temperature. Results obtained are as follows:
1. Nitrogen induces cyclic softening over the composition 0.267 to 0.496 wt % and influences the number of cycles to failure
2. Plastic strain range $\Delta\varepsilon_p$ is determined as a nonlinear function of both a number of cycles N_c and stress amplitude σ_a ($\exp(\sigma_a C3)$)
3. Radial arrangement of dendrites in cast steels has a significant influence on the fracture character
4. In tension as well as in low-cycle fatigue, the interstitial nitrogen promotes planar character of deformation.

1. Introduction

Nitrogen-alloyed austenitic steels and cast steels exhibit a variety of important properties such as high strength, ductility and fatigue crack resistance in aggressive environments (cf Foct and Hendry, 1989; Foct, 1990; Fila, 1987, 1992 and 1993; Fila and Zatorski, 1993). The effect of high content of nitrogen (above 0.25 wt %) on fatigue properties have been reported for special steels of extremely high Cr or Mn content, rather than for typical austenitic steels, because of the nitrogen solubility limit (cf Ishizaki and Mineura, 1988; Gavrilyuk and Efimenko, 1990). The dislocation arrangements formed in austenitic stainless steels during a low cycle fatigue processes at room, low and high temperatures, respectively, are strongly dependent on the nitrogen content (cf Takemoto et al., 1986; Kondoh et al., 1987; Taillard and Foct, 1988; Oda et al., 1990; Vogt et al., 1990). The aim of the present paper is to determine dynamic characteristics and microstructural behaviour of austenitic Cr-Ni-Mn-Mo-Nb-N cast steels during low-cycle fatigue process.

2. Materials and experimental procedure

2.1. Materials

The materials are LO3H21AN16G5M4Nb and L08H21AN16G5M4Nb cast irons and their chemical compositions are listed in Table 1. Primarily, they were designed to be identical except for their corbon content. Therefore, they were composed and made in an electric arc furnace and refined by VOD/VAD processes. Both the materials were remelted under industrial conditions by an induction melting under air atmopshere. The higher N, Mn and Cr contents were added in order to increase the nitrogen content beyond the solubility limit for a normal atmosphere.

Table 1. Chemical compositions and mechanical properties of Cr-Ni-Mn-Mo-Nb-N cast steels

No.	Chemical composition [%]								Mechanical propert.			
	C	N	Mn	Si	Cr	Ni	Mo	Nb	$\sigma_{0.2}$	T.S.	A5*	R.A.
1A	0.087	0.267	4.7	0.42	20.9	16.35	3.70	0.18	361	553	23.0	15.9
2A	0.085	0.358	3.9	0.49	21.7	16.36	3.68	0.18	373	444	5.6	11.2
3A	0.086	0.384	3.6	0.50	22.2	16.25	3.65	0.19	403	499	5.4	11.2
4A	0.082	0.400	3.2	0.49	23.0	16.09	3.60	0.17	364	405	3.0	7.2
5A	0.082	0.496	2.9	0.50	23.8	15.93	3.55	0.17	411	499	4.5	10.0
1B	0.089	0.290	4.8	0.39	21.2	16.37	3.70	0.16	334	484	4.9	14.4
2B	0.088	0.342	5.3	0.38	21.1	16.32	3.70	0.15	384	397	1.8	6.6
3B	0.087	0.408	5.9	0.38	20.9	16.25	3.68	0.16	342	437	5.3	12.1
4B	0.088	0.470	6.6	0.38	20.7	16.13	3.65	0.16	414	478	8.2	12.8
5B	0.094	0.494	7.2	.37	20.5	16.06	3.68	0.16	412	488	7.3	12.1
C1	0.024	0.290	4.2	0.42	19.5	15.59	3.06	0.17	328	525	19.9	28.4
C2	0.025	0.270	3.8	0.58	20.1	15.39	3.01	0.17	306	515	18.1	25.4
C3	0.027	0.280	3.6	0.70	20.6	15.21	2.98	0.17	316	568	15.8	22.5
C4	0.030	0.310	3.3	0.81	21.2	14.98	2.91	0.16	329	506	18.0	24.4
C5	0.031	0.330	3.0	0.91	21.8	14.73	2.85	0.15	354	455	9.1	23.8
D1	0.022	0.320	4.8	0.48	20.3	16.56	3.45	0.20	319	401	3.7	24.6
D2	0.022	0.320	5.4	0.60	20.1	16.40	3.45	0.20	341	496	11.7	23.0
D3	0.024	0.300	5.9	0.77	19.9	16.30	3.40	0.19	321	539	9.9	29.2
D4	0.023	0.270	6.7	0.84	19.6	16.16	3.36	0.19	321	487	10.6	22.0
D5	0.025	0.250	7.1	0.94	19.3	16.06	3.32	0.18	311	581	27.9	32.2
E	0.071	0.370	7.9	0.43	26.5	17.13	4.19	0.10	382	453	1.7	4.6

*A5 – Elongation of the specimens.

The cast irons were then bottom casted in sand castings into round bars, 25 mm in diameter and 150 mm in length, respectively. A few of A, B series bars were 10% cold-worked and then solution – treated at 1373 K for 1h. Specimens were machined with a reduced section of 10 mm in diameter and of 50 mm gauge length for tension tests, and of 10 mm gauge length for cyclic deformation tests. A few small specimens were cut out from these bars, aged at 923 K for 1h and air cooled (cf Fila, 1992 and 1993; Fila and Zatorski, 1993).

2.2. Tensile and low cycle fatigue tests

Fatigue and tensile tests were carried out under ambient conditions on the MTS 810.12 series (closed loop servo-hydraulic) materials testing machine of ±100 kN load capacity. All the tests were conducted at the constant strain rate $2.5 \cdot 10^{-3}$ s^{-1}. The strain was monitored by the MTS 832.11 C dynamic extensometer with a 10 mm gauge length which was suitable for measuring strains up to ±20%.

Fatigue tests were performed on hand-polished specimens in a block mode until complete failure under a sinusoidal stress $(R = -1)$ with a cyclic frequency 0.5 Hz.

2.3. Microstructural and fractographic observations

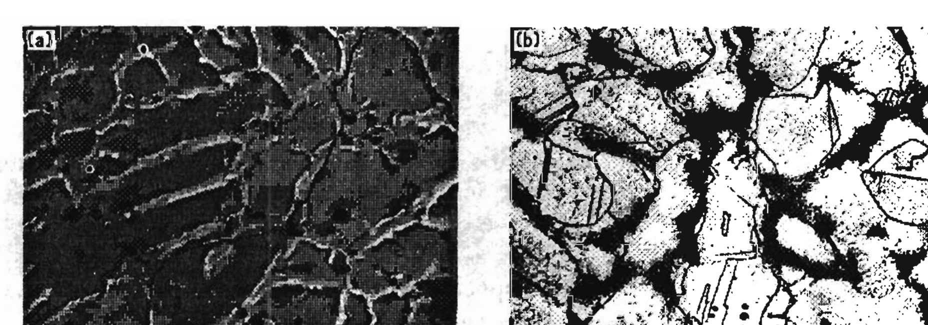

Fig. 1. Microstructure of 5A cast steel as-received (a) and annealed for 1h at 923 K after 10% cold-working and solution tretment for 1h at 1373 K (visible nitrides); magnification 630×

Optical metalography methods were used to reveal a structure of the received in the aforementioned way and heat treated specimens (see Fig.1). Specimens for miscrostructural observation were cut out from cyclically deformed specimens. The specimens were then sliced into 0.25 mm t × 3 mmϕ by spark errosion machine and thin foils were prepared by the twin-jet electropolishing technique. The electropolishing solution was a mixture of 10% $HClO_4$ and 90% CH_3COOH. Microstructures were observed under the conventional BS-540 Tesla type, 80 ÷ 120 kV transmission electron microscope (TEM). Fracture surfaces after static tension and low cycle fatigue specimens were observed under the BS-300 Tesla type scanning electron microscope (SEM).

3. Experimental results

3.1. Tensile properties

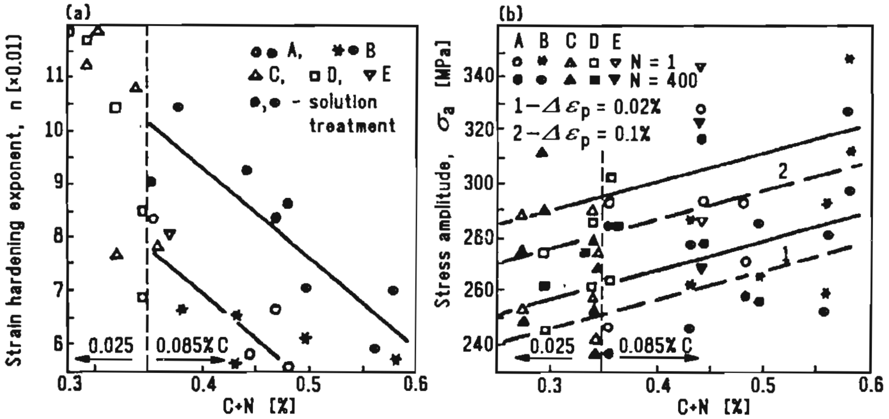

Fig. 2. Effect of C+N content on: (a) strain hardening exponent n at tensile tests, (b) stress amplitude σ_a at low-cycle fatigue

Mechanical properties of fatigue specimens are listed in Table 1. The Fig.2a shows the effect of C+N content on the strain hardening exponent n. The carbon contents in these specimens are almost the same (0.025% and 0.085%). Therefore, it is believed that the relations in Fig.2a show locally the effect of nitrogen on the exponent n. Although the same tendency was observed for A, B series, n-values for solution-treated specimens increase.

3.2. Low-cycle fatigue

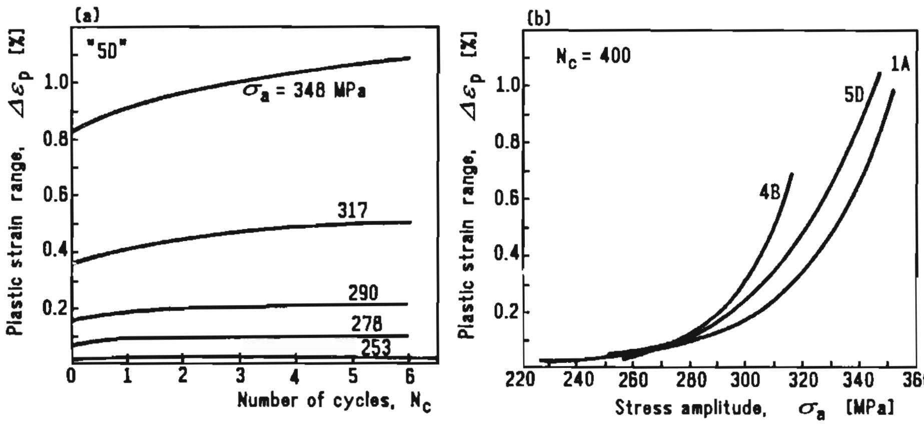

Fig. 3. Effect of number of cycles N_c (a) and stress amplitude σ_a (b) on plastic strain range $\Delta\varepsilon_p$ at low-cycle fatigue of cast steels: 5D, 4B and 1A

Changes in plastic strain range response $\Delta\varepsilon_p$, which depend on the number of cycles N_c and the stress amplitude σ_a are shown in Fig.3. The effects are summed up in the following equation (correlation coefficients above 0.9)

$$\Delta\varepsilon_p = C1\exp(C2\sigma_a)N^{C3} \quad [\%] \tag{3.1}$$

where

$C1, C2, C3$ – constants
σ_a – stress amplitude, [MPa]
N_c – number of cycles, $\Delta N_c = 500 \div 800$.

Constants calculated from Eq (3.1) are shown in Table 2. The σ_a stress amplitudes at $\Delta\varepsilon_p = 0.05\%$, $N_c = 1$ and $N_c = 400$ have been included in Table 2. The effects of interstitial C, N and substitutional Cr, Mn alloyed elements on $C2$, $C3$ values are shown in Fig.4. The effect of C+N content on the stress amplitude σ_a (at $N_c = 1$, $N_c = 400$, $\Delta\varepsilon_p = 0.02\%$ and $\Delta\varepsilon_p = 0.1\%$) is shown in Fig.2b.

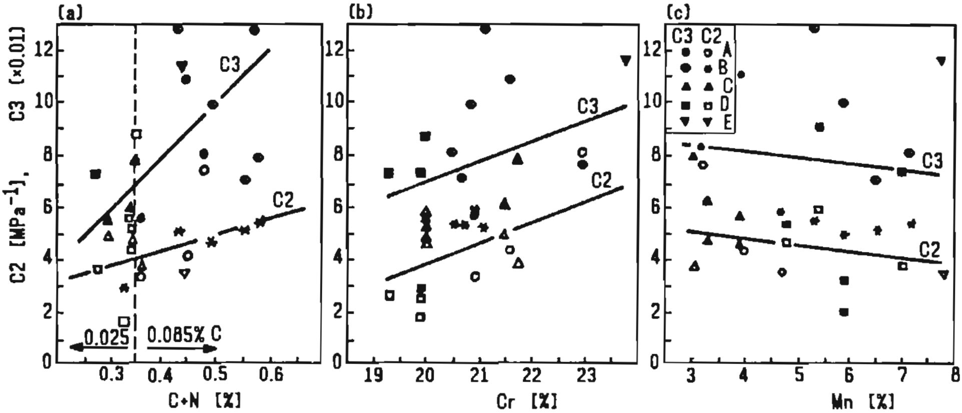

Fig. 4. Effect of C+N, Cr and Mn contents on the $C2$, $C3$ coefficients values in Eq (3.1)

Table 2. Coefficients in Eq (3.1) for high nitrogen cast steels after low cycle fatigue, $R = -1$, $f = 0.5$ Hz and $N_c = 500 \div 800$ cycles

Cast steel	σ_a [MPa]	$C1$ [%]	$C2$ ×0.01 MPa^{-1}	$C3$ ×0.01	C+N [%]	$\sigma_a - (\Delta\varepsilon_p = 0.05\%)$ $N_c = 1$ [MPa]	$N_c = 400$ [MPa]
1A	227÷342	5.14E-6	3.38	5.6	0.354	272	263
2A	283÷342	1.126E-7	4.13	10.8	0.443	313	301
4A	257÷297	3.633E-11	7.40	8.0	0.482	282	270
2B	249÷296	3.7E-8	5.07	13.2	0.430	276	258
3B	220÷284	9.0E-8	4.68	9.8	0.495	281	273
4B	257÷316	5.0E-8	5.07	7.0	0.558	277	267
5B	296÷349	1.6E-9	5.24	8.0	0.582	331	316
2C	253÷323	7.0E-7	4.42	5.4	0.295	260	247
4C	264÷323	1.61E-7	4.75	6.0	0.340	275	262
5C	285÷354	1.85E-6	3.73	7.7	0.361	289	-
1D	243÷354	4.5E-7	4.50	5.14	0.344	261	254
2D	252÷291	5.6E-9	5.74	8.86	0.345	270	266
3D	317÷418	1.85E-4	1.76	2.95	0.324	338	317
5D	310÷386	2.71E-6	3.63	7.2	0.275	273	258
E2	213÷349	5.2E-7	3.59	11.3	0.441	314	299

3.3. Changes in microstructure and fractography

Photographs given by Fila and Zatorski (1993) – Fig.8 and Fig.9 – represent the microstructure of cast steels after tensile test: (a) shows a celluar type of dislocation structure in a specimen of no nitrogen content, and (b) shows a

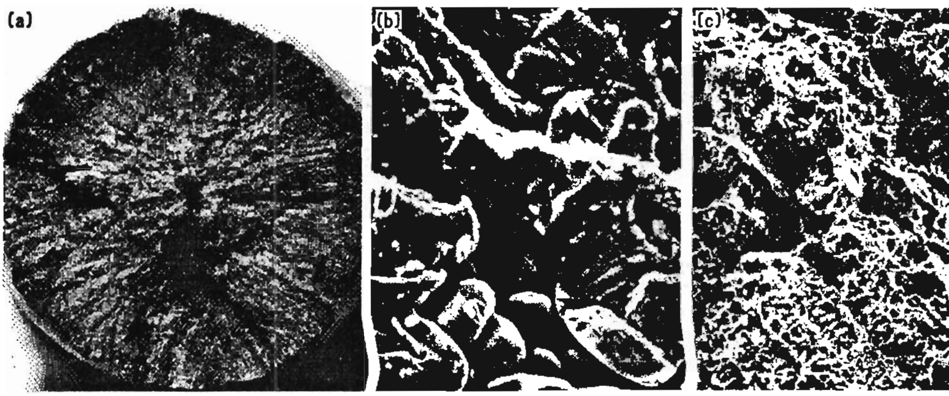

Fig. 5. Scanning electron micrographs of the fracture surfaces of 5B (a) and E2 (b), (c) specimens illustrating the sites where fatigue cracking initiated and the effect of radial dendrite arrangement. SEM, magnification: (a) – 7×, (b) – 1000×, (c) – 150×

planar type of dislocation structure in a specimen with 0.4% nitrogen content. Photographs in Fig.6 present the changes in microstructure of cast steels failed after low cycle fatigue. In fatigue tests of cast steels, cracks appeared either inside or on the outer surface of specimens. Characteristic example of the inner crack is illustrated in Fig.5.

4. Discussion

4.1. The effect of nitrogen on tensile properties

As shown in Table 1, cast steels with lower nitrogen and carbon contents display higher ductility at almost invariant strength in all cases. The dendritic segregation effect of nitrogen which is observed in a microstructure after annealing at 650°C/h and on fracture surfaces (cf Fila and Zatorski, 1993 – Fig.1b) can be responsible for that. Strain hardening exponents indicate higher values after the solution treatment. This indicates a decrease in dendritic segregation and softening role of higher nitrogen contents. Celular type of the dislocation structure is formed in specimens with no nitrogen content and a planar structure is formed in specimens with a high nitrogen content (cf Fila and Zatorski, 1993 – see Fig.8 and Fig.9). The stacking fault energy (SFE) is higher in the former ones.

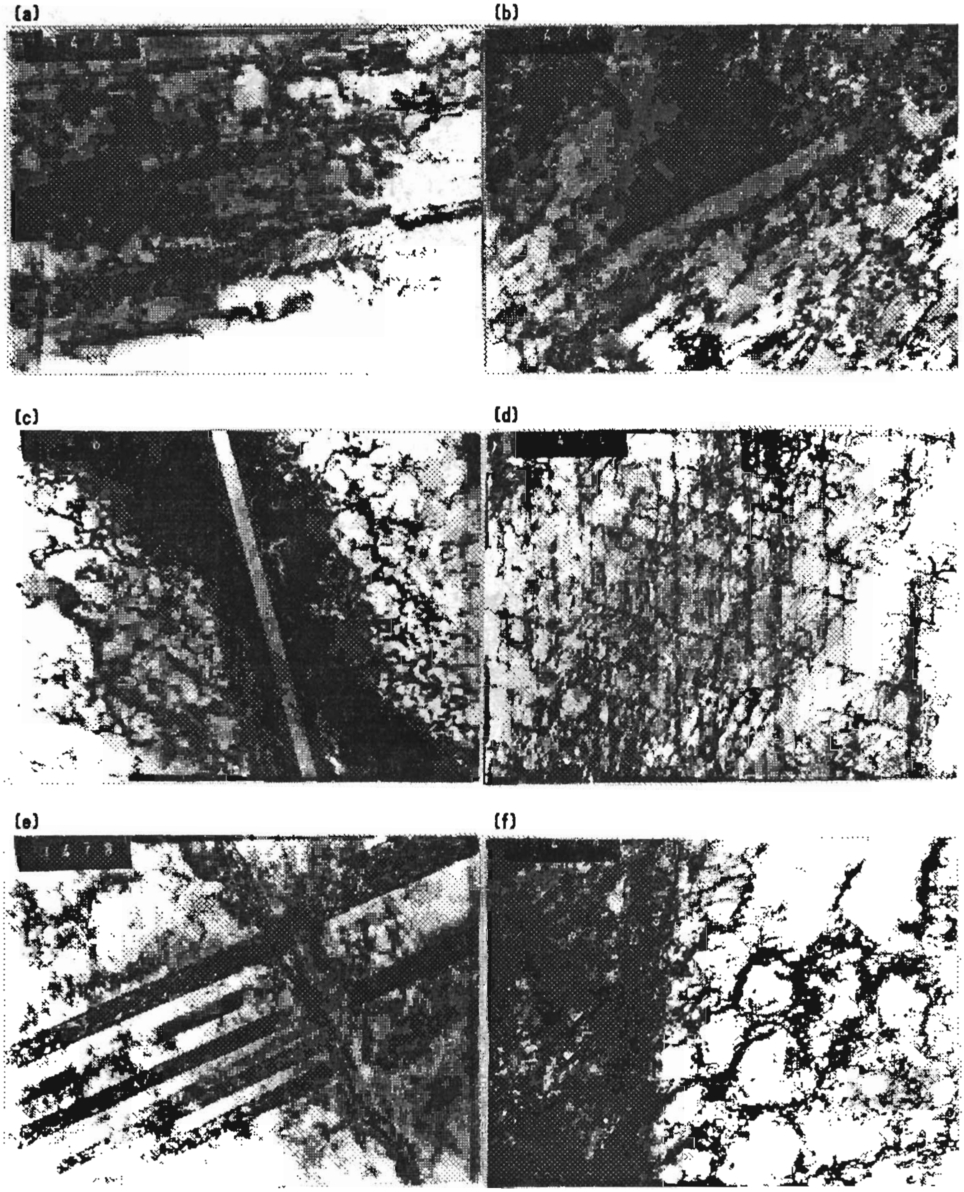

Fig. 6. Dislocations arrangement in Cr-Ni-Mn-Mo-Nb-N cast steels fatigued at room temperature at cyclic frequency 0.5 Hz: (a), (b) 5B, planar – (c), (d) 1A, cellular and planar – (e), (f) cellular type of dislocation structure. TEM, magnification 35000×

4.2. The effect of nitrogen on low cyclic behaviour of cast steels

Low cycle behaviour of cast steels is described by Eq (3.1) and showed in Fig.2b and 3. In contrast to steels, when the cast steels are subject to cyclic load at room temperature, neither cyclic hardening to saturation nor cyclic hardening at early stage of deformation takes place (cf Takemoto et al., 1986; Vogt et al., 1990).

The values of $C2$ and $C3$ coefficients are more sensitive to the chromium content than to the manganese one (Fig.4). Alloying elements affect the distribution of carbon and nitrogen atoms in austenite in different ways. Nickel, increasing the chemical potential of carbon in iron compounds, causes inhomogeneous short range atomic ordering. Manganese, decreasing the thermodynamical activity of carbon, assists decomposition of the solid solution with the formation of manganese-carbon clusters with a very low content of iron and intercluster regions depletion in manganese and carbon. But, in iron-nitrogen austenite of an even greater content of manganese(about 10 wt %) causes only redistribution of nitrogen atoms without decomposition of the solid solution (cf Foct, 1990; Gavrilyuk and Efimenko, 1990).

Interstitial atoms gather around chromium atoms to form a kind of interstitial-subsitutional (I-S) complexes such as a short range ordered structure. Silicon probably promotes formation of the I-S complexes around chromium atoms. Low cycle fatigue softening, which proceedes commonly in Cr-Ni and Cr-Mn austenitic steels strengthened by both carbon and nitrogen, is thought to be the result of breakdown of the I-S complexes (cf Kondoh et al., 1987; Oda et al., 1990).

It is well known that the cyclic behaviour of FCC metals and alloys depend strongly on their stacking fault energies (SFE). The cross slip of dislocations is easier for higher SFE, which increases the interaction between dislocations, resulting from the work hardening (cvf Takemoto, 1986). The decrease of strain hardening coefficients with the nitrogen content coincides with transition from cellular to planar distribution of dislocations. Nitrogen inhibits cross-slip of dislocations, mainly through the pinning effect, and promotes a planar distribution of dislocations. However, most of the measurements of SFE show that N does not influence SFE or lowers it a little (cf Taillard and Foct, 1988; Vogt et al., 1990). Thus, it can be postulated that the planar distribution of dislocations is attributed to short range ordered zones (SRO-zones) formed by Fe-Cr-Mo and N and their interactions with dislocations (cf Takemoto et al., 1986; Ishizaki and Mineura, 1988; Taillard and Foct, 1988; Vogt et al., 1990). The planar mode of deformation is self-evident in the high-nitrogen cast steel, Fig.6a,b. In contrast, cellular structure of dislocation is observed in low nitrogen and low carbon cast steel Fig.6e,f. In addition, it is worth noticing that the transition from planar to cellular character of deformation is caused by the low cycle softening, as illustrated in Fig.6c,d. Fractographic observations of fatigue fractures indicate the notch effect caused by the dendrite distribution nearly an axis of the specimens from nitrogen cast steels (cf Fila and Zatorski, 1993 and in Fig.5). This harmful effect was not observed in lower C+N alloyed cast steels.

5. Conclusions

The efect of nitrogen on mechanical properties and low cycle behaviour of Cr-Ni-Mn-Mo-Nb-N austenitic stainless cast steels has been investigated at room temperature. The major conclusions drawn from this study are that:

- N content from 0.267 to 0.496 wt % induces cyclic softening over the whole lifetime
- Plastic strain range $\Delta\varepsilon_p$ is determined as a function of both the number of cycles N_c and stress amplitude σ_a
- Radial arrangement of dendrites in cast steels has a significant influence on the fracture character
- In static tension as well as at low-cycle fatigue, the interstitial nitrogen promotes planar type of dislocation structure.

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Wpływ azotu na mechanizm małocyklowego zmęczenia austenitycznych staliw nierdzewnych

Streszczenie

Przebadano wpływ azotu ($0.267 \div 0.496\%$) na własności mechaniczne i niskocyklowe zmęczenie austenitycznych staliw nierdzewnych Cr-Ni-Mn-Mo-Nb-N. W wyniku badań stwierdzono, że:

1. wysoka zawartość azotu sprzyja cyklicznemu zmiękczaniu staliw austenitycznych i ujawnianiu planarnego charakteru struktury dyslokacyjnej;

2. przy niższych zawartościach azotu występuje komórkowy lub mieszany typ zmęczeniowej struktury dyslokacyjnej;
3. promieniowy układ dendrytów i efekt osiowych karbów skurczowych w odlewach staliw ma znaczący wpływ na ich własności i charakter przełomu;
4. zakres odkształceń plastycznych $\Delta\varepsilon_p$ jest określony jako funkcja zarówno liczby cykli N_c jak i amplitudy naprężenia σ_a.

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