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Effects of carbon content on microstructures and magnetic properties of annealed or solution treated Fe-Cr-Ni-C alloys

S. YOKOYAMA, T. INUI
Metallurgical Research Laboratory, Hitachi Metals, Ltd., 2107-2 Yasugi-cho Yasugi, Shimane 692-8601, Japan
E-mail: shinichiroh_yokoyama@hitachi-metals.co.jp

Y. MINAMINO
Department of Adaptive Machine Systems, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871, Japan

The correlations between microstructures and magnetic properties were studied in four Fe-17.5 mass%Cr-2.0 mass%Ni-Xmass%C ($X = 0.3$–0.6) alloys. Each alloy consisted of ferromagnetic α and $M_{23}C_6$ carbide phases by step-annealing at 1053 K and 848 K. The number of the $M_{23}C_6$ carbide particles increased with the carbon content, and this microstructural variation caused a deterioration of the soft magnetic properties. On the other hand, the alloy with 0.3 mass% carbon content consisted of ferromagnetic α' and paramagnetic retained γ structures after solution treatment at 1473 K. The amount of the stable γ structure increased with the carbon content, while the amount of the α' structure decreased. This microstructural variation caused a decrease in the relative permeability, μ_r, and the stabilization of the paramagnetic property to low temperatures below room temperature. The temperature stability of the μ_r values was closely related to the martensite start temperature, M_s. From an equation for the estimation of M_s from the chemical compositions of the γ phase, the M_s's of the alloys with 0.3, 0.4, 0.5 and 0.6 mass%C were estimated to be 326, 289, 241 and 216 K respectively. These values were consistent with the M_s's expected from the microstructures and temperature dependences of the μ_r values. © *2003 Kluwer Academic Publishers*

1. Introduction

Some Fe-based alloys have both ferromagnetic α and paramagnetic γ phases [1–3]. For example, an Fe-17.5 mass%Cr-0.5 mass%C alloy, which is fully annealed at temperatures below the A_1 temperature, consists of ferromagnetic α and carbide phases and exhibits soft magnetic property. On the other hand, this alloy mainly consists of paramagnetic γ phase by solution treatment at higher temperatures above the A_3 temperature [1]. The combination of these heat treatments enables us to produce varying amounts of both ferromagnetic and paramagnetic phases in industrial iron metal parts with mono chemical composition. This enables the production of magnetic circuits without the need to bond the ferromagnetic and paramagnetic parts. Such a component would be structurally highly reliable because a bonding interface is a region of weakness sometimes leading to breakdown, and would be easily recycled because they consist of an alloy with mono chemical composition [3].

The temperature stability of the paramagnetic property is required in the practical use of the parts for applications such as the magnetic circuits because the paramagnetic property of the γ phase is lost by the transformation from γ phase to the ferromagnetic α' phase below the martensite start temperature, M_s. Therefore, a low M_s of the alloy is desired to obtain more stable paramagnetic property. In a previous study [1], the effects of nickel content on the M_s and stability of the paramagnetic property in four Fe-17.5 mass%Cr-Z mass%Ni-0.5 mass%C ($Z = 1.0$–2.4) alloys were clarified, and it was found that more than 2.0 mass%Ni content was necessary to preserve the stable paramagnetic γ phase to temperatures below room temperature.

Many metallographic studies have been done on the effects of carbon content in ternary Fe-Cr-C alloys [4–11]. In addition, a lot of studies on the effect of carbon content on M_s have been reported in Fe-Cr-C and other carbon containing alloys [7, 10, 12–16]. It is well-known from these studies that carbon in the γ phase has great influence on the M_s. However, few studies have been carried out on the magnetic properties and the correlation between microstructures and magnetic properties even in ternary Fe-Cr-C alloys [2, 17]. Especially, the effect of carbon content on the temperature stability of the paramagnetic property has not been reported yet. In addition, the relation between microstructures and soft magnetic properties has not been studied, although both microstructures and soft magnetic properties of the annealed alloys are expected to vary with the carbon content. That is to say, the effects of carbon content on microstructures and magnetic properties of the Fe-17.5 mass%Cr-2.0 mass%Ni-0.5 mass%C alloy have not been studied yet.

It is practically and scientifically important to elucidate the correlation between microstructures and magnetic properties in alloys with different carbon content. Therefore, the purpose of this study is to clarify the effects of carbon content on microstructures and magnetic properties of annealed or solution treated Fe-17.5 mass% Cr-2.0 mass%Ni-X mass%C alloys ($X = 0.3$–0.6).

2. Experimental procedures

Four Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloy ingots were prepared by vacuum induction melting. Their chemical compositions are listed in Table I. The ingots were homogenized at 1493 K for 36 ks, and then slowly cooled in the furnace in the air. These homogenized ingots were hot forged, and then hot rolled to the sheets of 5 mm in thickness at 1273 K. These hot rolled sheets were step-annealed for softening in argon atmosphere through the heat treatment in Fig. 1a, and then cold rolled to the sheets of 1 mm in thickness.

For the phase analysis and observation of the microstructures, samples of 1 mm × 10 mm × 15 mm in size were cut out from the cold rolled sheets. In addition, ring samples of 1 mm in thickness, 33 mm in inner diameter and 45 mm in outer diameter were also cut out for the measurement of the magnetic properties. Both samples were step-annealed in argon atmosphere as shown in Fig. 1b. The phases were identified by X-ray diffractometry. The microstructures were observed using a scanning electron microscope (SEM). The magnetic properties were measured with a DC B-H analyzer.

Samples of 1 mm × 10 mm × 15 mm and 1 mm × 20 mm × 20 mm in size were also cut out from the same cold rolled sheets for the observation of the microstructures and the measurement of magnetic property respectively. Both samples were solution treated in vacuum furnace as shown in Fig. 1c, then bathed in ethanol and kept at each temperature in the range from 233 to 273 K for 0.6 ks. Their microstructures were observed using an optical microscope. The relative permeability, μ_r, was measured with a permeability meter.

TABLE I Chemical compositions of the samples (mass%)

Alloy no.	C	Si	Mn	Ni	Cr	Fe
1	0.31	0.18	0.51	1.96	17.59	Bal.
2	0.41	0.18	0.52	1.96	17.68	Bal.
3	0.51	0.18	0.52	1.97	17.67	Bal.
4	0.61	0.18	0.52	1.97	17.72	Bal.

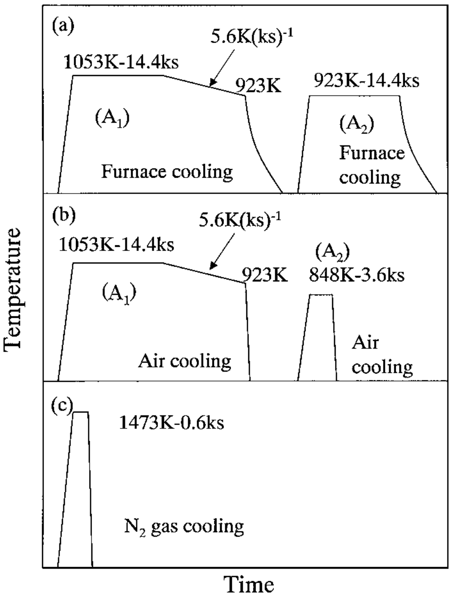

Figure 1 Schematic diagrams for the heat treatments: (a) annealing pattern to soften the samples, (b) annealing pattern to test the samples and (c) solution treatment for the samples.

3. Results and discussion

Figs 2 to 4 show the X-ray diffraction patterns, microstructures and the DC magnetic properties of Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$ to 0.6) alloys step-annealed in argon atmosphere in the condition shown in Fig. 1b, respectively. As shown in Fig. 2, each alloy consists of α-phase and $M_{23}C_6$ carbide. The intensity of the $M_{23}C_6$ carbide becomes stronger with increasing carbon content. Although the size and shape of the carbides are almost independent of the carbon content (Fig. 3), it should be noted that the number of the $M_{23}C_6$ carbides increases with the carbon content. As for the magnetic properties (Fig. 4), the soft magnetic properties deteriorate slightly

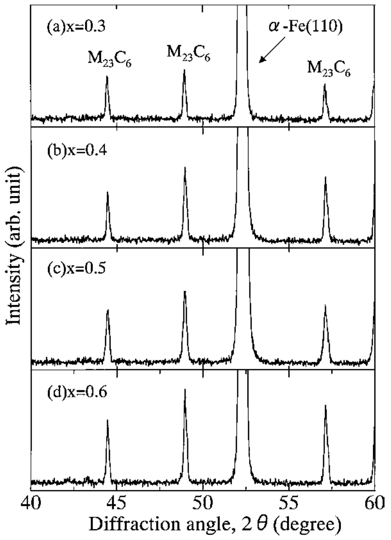

Figure 2 X-ray diffraction patterns of the annealed Fe-17.5 mass% Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys.

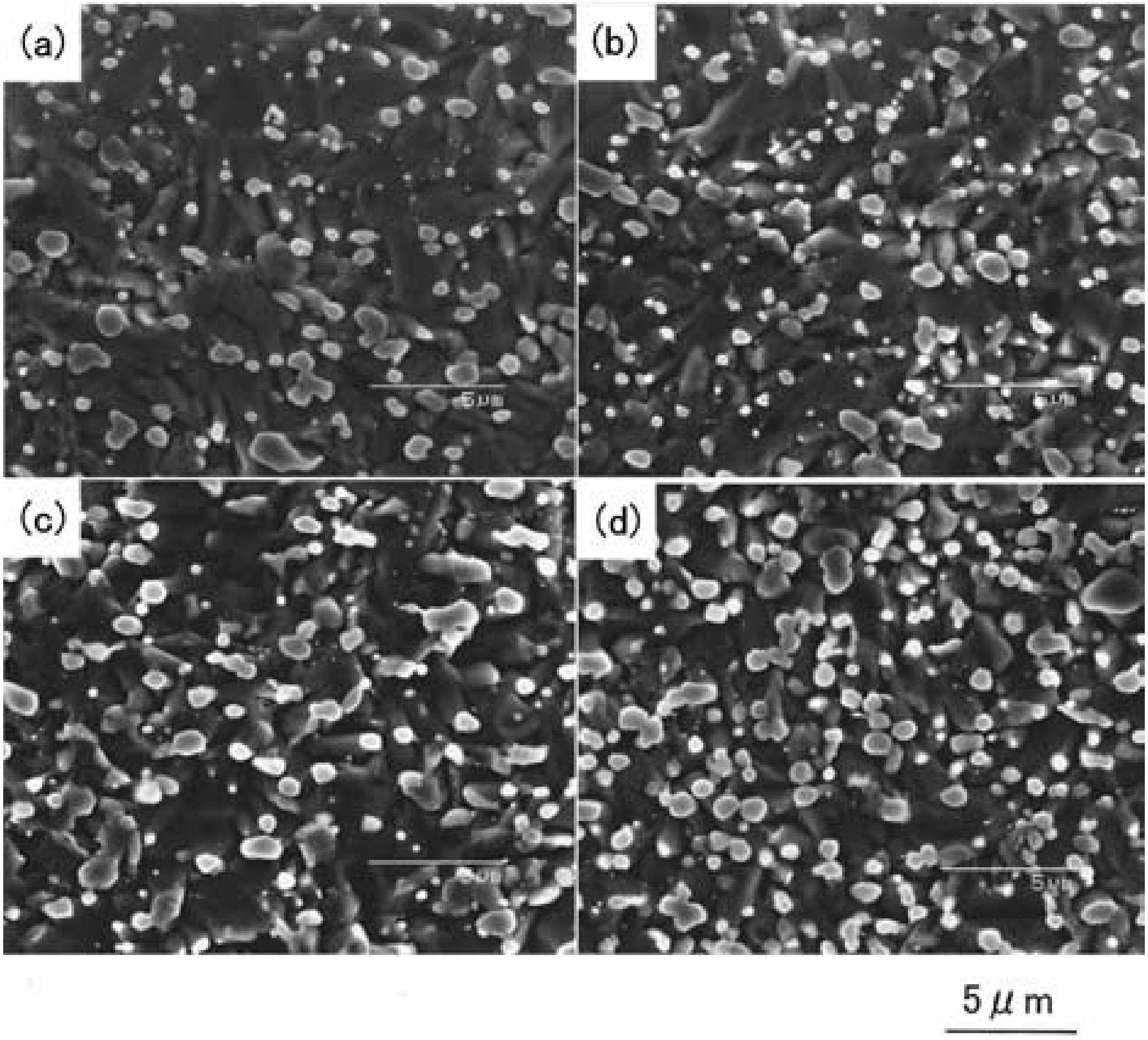

Figure 3 Microstructures of the annealed Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys: (a) $X = 0.3$, (b) $X = 0.4$, (c) $X = 0.5$ and (d) $X = 0.6$.

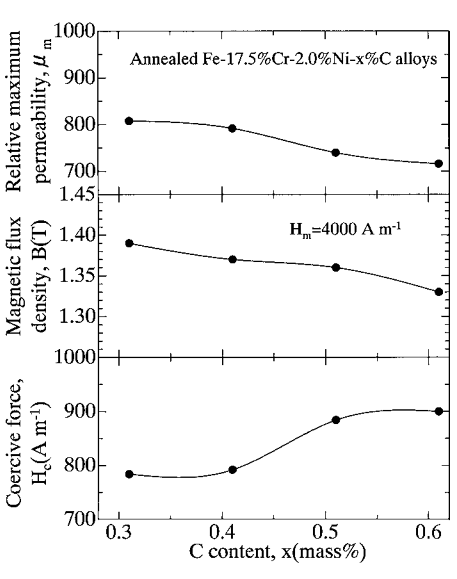

Figure 4 Carbon content dependence of the DC magnetic properties of the annealed Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys.

with the increase of the carbon content; the relative maximum permeability, μ_m, and the magnetic flux density, B, at 4000 Am^{-1} decrease slightly with increasing carbon content, while the coercive force, H_c, increases. In general, the precipitates or inclusions in bulk soft magnetic alloys obstruct the rotation of magnetic domain and the migration of domain wall for soft magnetic properties. Therefore, it is considered that this deterioration of the soft magnetic properties with the increase of the carbon content is mainly due to the increase in the number of fine $M_{23}C_6$ carbide particles.

Fig. 5 shows the microstructures of the alloys after solution treatment of Fig. 1c, and those of the alloys after the solution treatment and cooling to 233 K (referred to as "subzero treatment"). A martensitic (α') structure is observed in the alloys of 0.3 mass%C in both the solution treated (Fig. 5a) and subzero treated (Fig. 5b) conditions, although some retained austenite (γ) is also observed. On the other hand, a stable retained γ structure covers the alloys of 0.4 mass%C (Fig. 5c and d); α' is scarcely observed in the solution treated

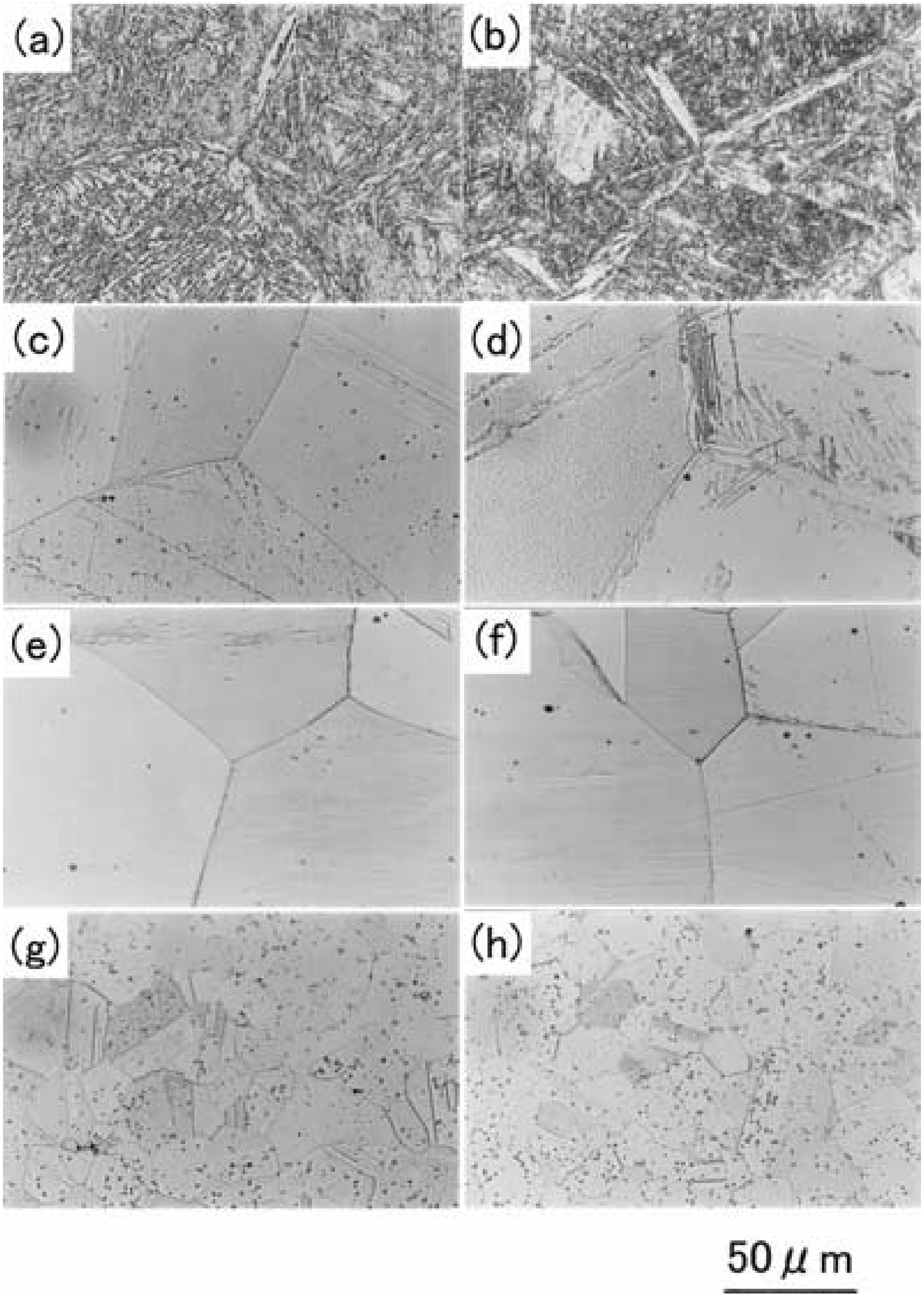

Figure 5 Microstructures of the Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C (a) and (b): $X = 0.3$, (c) and (d): $X = 0.4$, (e) and (f): $X = 0.5$, (g) and (h): $X = 0.6$) alloys. (a)(c)(e)(g): solution treated at 1473 K, and (b)(d)(f)(h): solution treated at 1473 K and then subzero treated at 233 K.

alloy (Fig. 5c), while a small amount of α' exists near the grain boundaries in the subzero treated condition (Fig. 5d). The $\gamma \rightarrow \alpha'$ transformation in the alloys of 0.3 and 0.4 mass%C occurs during the cooling process from 1473 K to room temperature or to 233 K. This indicates that the martensite start temperatures, M_s's, of the 0.3 and 0.4 mass%C alloys exist above the room temperature or in the range between room temperature and 233 K, respectively. In the alloys of 0.5 mass%C with both treatments (Fig. 5e and f), the structure is only γ phase, which is quite stable even at low temperature of 233 K. On the other hand, the higher carbon alloys of 0.6 mass%C (Fig. 5g and h) in both the solution treated and subzero treated, exhibit the microstructure of γ phase with many insoluble carbides. The grain size of γ phase is small compared with that of the alloy of 0.5 mass%C, because a lot of fine carbides impede the grain growth of the γ phase.

Fig. 6 shows the relative permeability, μ_r, of various alloys after solution treatment of Fig. 1c. The μ_r sharply decreases with increasing carbon content

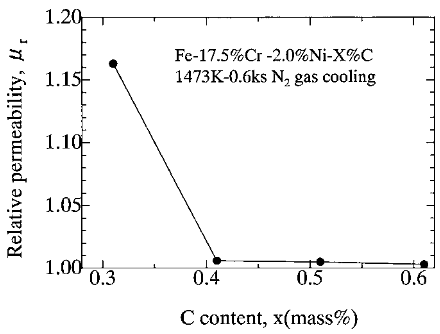

Figure 6 Carbon content dependence of the relative permeability of the solution treated Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys.

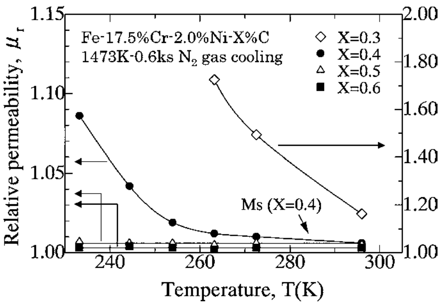

Figure 7 Carbon content dependence of the temperature stability of the relative permeability of the solution treated Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys.

up to 0.4 mass%C, and then slightly decreases for carbon content over 0.4 mass%C. Fig. 7 shows the temperature stability of the μ_r values below room temperature. The considerably higher μ_r values of the alloy of 0.3 mass%C than the others are due to the α' phase which is the major phase in the alloy (Fig. 5a); the α' phase has higher μ_r values than the γ phase. The μ_r values for lower carbon alloys of 0.3 mass%C and 0.4 mass%C increase with decreasing temperatures, while the higher carbon alloys of 0.5 mass%C and 0.6 mass%C exhibit no increase in the μ_r. The increase in the μ_r values is closely related to the increasing amounts of α' phase due to the subzero treatments, and the increase of the α' phase due to the subzero treatment is clearly observed especially in the 0.4 mass%C alloy (Fig. 5c and d). It is also found that the M_s of the 0.4 mass%C alloy is in the range from 273 to 296 K where the μ_r begins to increase. On the other hand, the constant μ_r values of higher carbon content alloys (0.5 and 0.6 mass%C) even at low temperatures from 293 to 233 K is due to the microstructures consisting of stable retained γ phase or γ phase with many carbides as shown in Fig. 5e–h. The α' phase is not observed in these alloys and hence the M_s's of alloys with carbon content greater than 0.5 mass%C are below 233 K.

Fig. 8 shows the equilibrium phase diagram of the Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0$–1.5) alloy calculated by Thermo-Calc. Table II lists the equilibrium phases and their chemical compositions of the γ phase for each alloy at 1473 K calculated by Thermo-Calc. Table II also shows the M_s temperatures evaluated from the calculated chemical compositions of the γ phase and the following equation for estimating M_s [7, 12].

TABLE II Equilibrium phases and chemical compositions of the γ phase at 1473 K calculated by Thermo-calc., and estimated M_s temperatures

Alloy no.	Phases (1473 K)	Chemical compositions of the γ phase (mass%)	Estimated M_s (K)
1	$\gamma + \alpha$	Fe-17.08%Cr-2.05%Ni-0.35%C	326
2	γ	Fe-17.68%Cr-1.96%Ni-0.41%C	289
3	γ	Fe-17.67%Cr-1.97%Ni-0.51%C	241
4	$\gamma + M_7C_3$	Fe-17.47%Cr-1.98%Ni-0.57%C	216

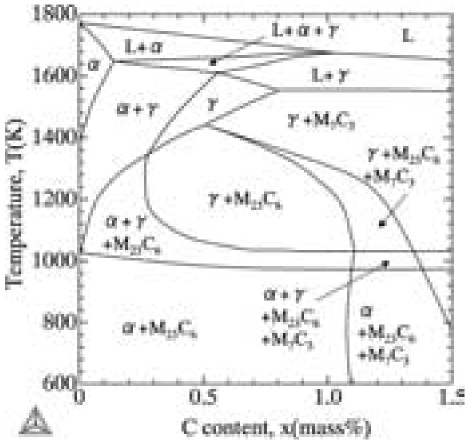

Figure 8 Phase diagram of the Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0$–1.5) alloy calculated by Thermo-calc.

$$M_s(\mathrm{K}) = 834\text{-}474 \times (\text{mass\%C})\text{-}33 \times (\text{mass\%Mn})\text{-}17 \times (\text{mass\%Ni})\text{-}17 \times (\text{mass\%Cr})$$

In this estimation, it was assumed that manganese dissolved completely in the γ phase. As shown in Fig. 2, the existence of α and $M_{23}C_6$ phases in annealed alloys is consistent with the calculated phase diagram (Fig. 8). The M_s of the 0.3 mass%C alloy is estimated to be 326 K as listed in Table II. This M_s is consistent with the microstructure of the alloy with 0.3 mass%C being almost fully α' phase (Fig. 5a). Moreover, the M_s's of the alloys with 0.4, 0.5 and 0.6 mass%C are estimated to be 289, 241, and 216 K respectively. These are almost consistent with the M_s's estimated from the temperature dependences of the μ_r values shown in Fig. 7. Thus, the microstructures and the magnetic properties are closely related to each other, and the increase in carbon content is effective in lowering μ_r and stabilizing the paramagnetic property to low temperatures.

4. Conclusions

1. Four Fe-17.5 mass%Cr-2.0 mass%Ni-X mass%C ($X = 0.3$–0.6) alloys, consisting of ferromagnetic α and $M_{23}C_6$ carbide phases, were produced by step-annealing at 1053 and 848 K. The number of the $M_{23}C_6$ carbide particles increased with the carbon content. This microstructural variation caused a deterioration of the soft magnetic properties.

2. The alloy with 0.3 mass% carbon content consisted of ferromagnetic α' and paramagnetic retained γ structures after solution treatment at 1473 K. The amount of the stable γ structure increased with the carbon content, while the amount of the α' structure decreased. The γ structures were stable even after subzero treatment at 233 K in the alloys with carbon content greater than 0.5 mass%. These stable γ structures caused a decrease in the relative permeability, μ_r, and stabilized the paramagnetic property to 233 K.

3. The temperature stability of the μ_r values was closely related to the martensite start temperature, M_s. From an equation for estimating M_s from the chemical compositions of the γ phase, the M_s's of the alloys with 0.3, 0.4, 0.5 and 0.6 mass% carbon content were estimated to be 326, 289, 241 and 216 K respectively. These values were consistent with the M_s's expected from the microstructures and temperature dependences of the μ_r values.

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