

Effect of annealing temperature on the magnetic behaviour of Ni-rich permalloy magnetic materials

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Magnetic properties have been investigated in Ni-rich permalloy (Ni $\cong$ 80%) material in which process conditions have been controlled under hydrogen annealing by varying holding time, cooling rate and annealing temperature. The material thus prepared has been characterized for various magnetic parameters under different annealing profile conditions. The results indicate that Ni-rich permalloy material shows better magnetic properties at 300 Hz over 1 kHz under controlled conditions.

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There has been considerable research work going on for the development of magnetic materials for storage devices and audio head recording applications. Ni-Fe alloys are special class of magnetic materials based on the face-centered cubic crystal structure of Ni-Fe system. They usually contain 40% -90% Ni, up to a few per cent each of other alloying elements like Cu, Mo, Cr, Co, Mn and balance is Fe¹. Their remarkable magnetic properties have made them the subject of intensive study over the last about 40 years. As knowledge about these materials and their properties grew, so has the range of their applications broadened with time. The 76-80% Ni-Fe alloys or permalloys have been widely used in various applications since 1920².

With suitable alloying elements and proper control of processing conditions, the magnetic properties of the material can be controlled over wide range³. The doping of certain elements to the basic 80:20 nickel-iron alloy, like Mo, Cu, Cr and Mn have significant effect on the magnetic properties⁴⁻⁶. Both the binary and modified alloys are dependent on the final heat treatment. Apart from the purification and strain relieving aspect of the heat treatment, this effect is associated with the formation of the Ni₃Fe super lattice structure in the binary alloy. A similar structural change appears to occur in the modified alloy. The magnetic properties of permalloy depend

on annealing temperature, cooling rate and holding time^{7,8}. For any application, the rational selection of composition and processing procedure requires a comprehensive understanding of the relationship between engineering properties and processing in terms of metallurgical factors and fundamental magnetic properties of the material.

In this paper, we report on the performance of Ni-rich permalloys in terms of their magnetic properties under H₂ annealing by varying physical parameters like field strength, annealing temperature, cooling rate and holding time.

Experimental Procedure

Ni-rich permalloy material of thickness about 0.30 mm was investigated for the study. The elements and composition of the alloy were determined by wet chemical analysis and atomic absorption spectrophotometer as given in Table 1.

The samples were prepared in ring shape having outer and inner diameters of about ~30 mm and ~25 mm, respectively. These were annealed in H₂ atmosphere in the temperature range of 1100-1180°C under different processing parameters like variable holding time, cooling rate and annealing temperature. A block diagram of the experimental set-up⁹ used for annealing of permalloy material is given in Fig. 1.

The different annealing profiles performed on the samples to study the effect of annealing temperature, holding time and cooling rate are given in Table 2.

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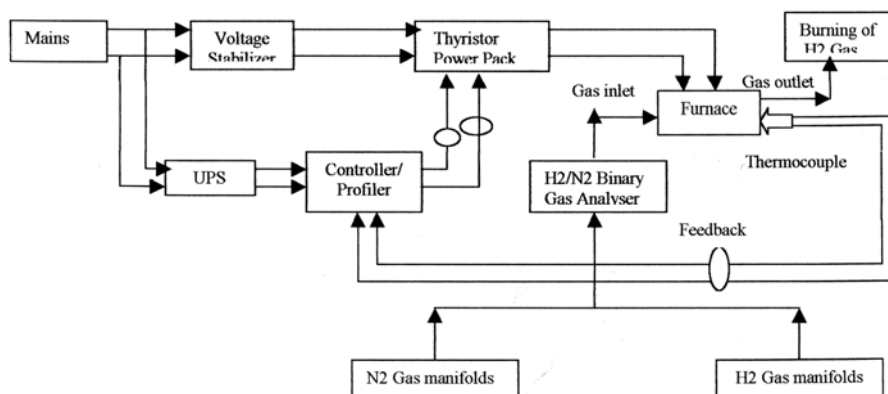


Fig. 1—Block diagram of the experimental set-up

Table 1—Composition of the permalloy material used in present study

Element	Content (wt%)
Ni	79.90
Fe	14.47
Mo	4.90
Mn	0.41
Cu	0.05
Cr	0.10
Co	0.05
C	0.01
Si	0.10
S	0.005
P	0.005

Table 2—Annealing Profiles of the Permalloy

Annealing temperature (°C)	Holding time (h)	Cooling rate (°C/min)
1100, 1120, 1140, 1150, 1160, 1180	2	2.5
1150	1, 2, 3	5.0
1150	2	2.0, 2.5, 3.0, 5.0, 7.0

For the magnetic measurements, toroid shape samples were prepared by stacking 5 number of rings and using 20 numbers of primary and secondary turns as shown in Fig. 2. The magnetic measurements under different processing parameters were carried out using B-H Analyser (AMH-401, Walker Scientific, USA). The magnetic properties such as induction, remenence, core loss, coercivity and peak permeability have been evaluated at 300 Hz and 1 kHz frequency by varying the magnetic field.

Results and Discussion

Induction (B)

A typical behaviour of induction in Ni-rich sample corresponding to a function of field strength, annealing temperature, cooling rate and holding time at 300 Hz and 1 kHz frequency is given in Fig. 3 (a-h)

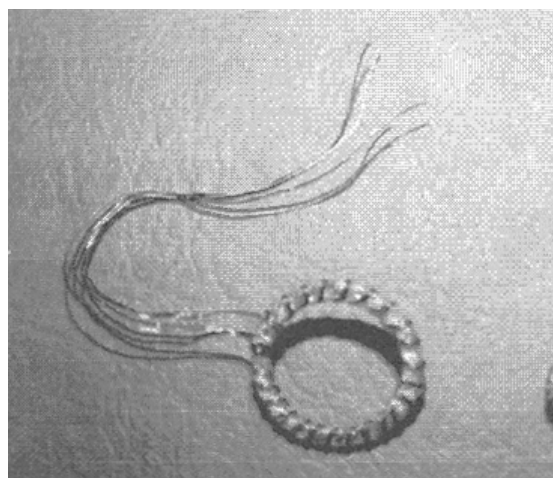


Fig. 2—Assembled toroid shaped sample

respectively. We observed that at input frequency of 300 Hz, induction increases exponentially from 620 mT to about 750 mT as field strength increases from 50 A/m to 300 A/m and thereafter tends to saturate at 1150°C. Similar behaviour is depicted at another temperature as shown in Fig. 3a. The temperature dependence of B also shows an increase up to 1140°C and thereafter within 10°C temperature range (i.e. 1140-1150°C), the induction shows a partial decrease but increases thereafter. This may be attributed to the change in the direction of magnetization within this temperature range (Fig. 3b). These measurements have been taken by cooling the material @ 2.5°C/min and by holding the sample for 2 h in order to attain stability with respect to magnetic domains within the samples. The B shows an interesting variation as a function of cooling rate in the samples. These results are reflected in Fig. 3c at a constant temperature 1150°C for holding time up to 2 h. It is observed that at a relatively lower field strength (i.e. 100 A/m) the variation in B is of larger magnitude whereas increase

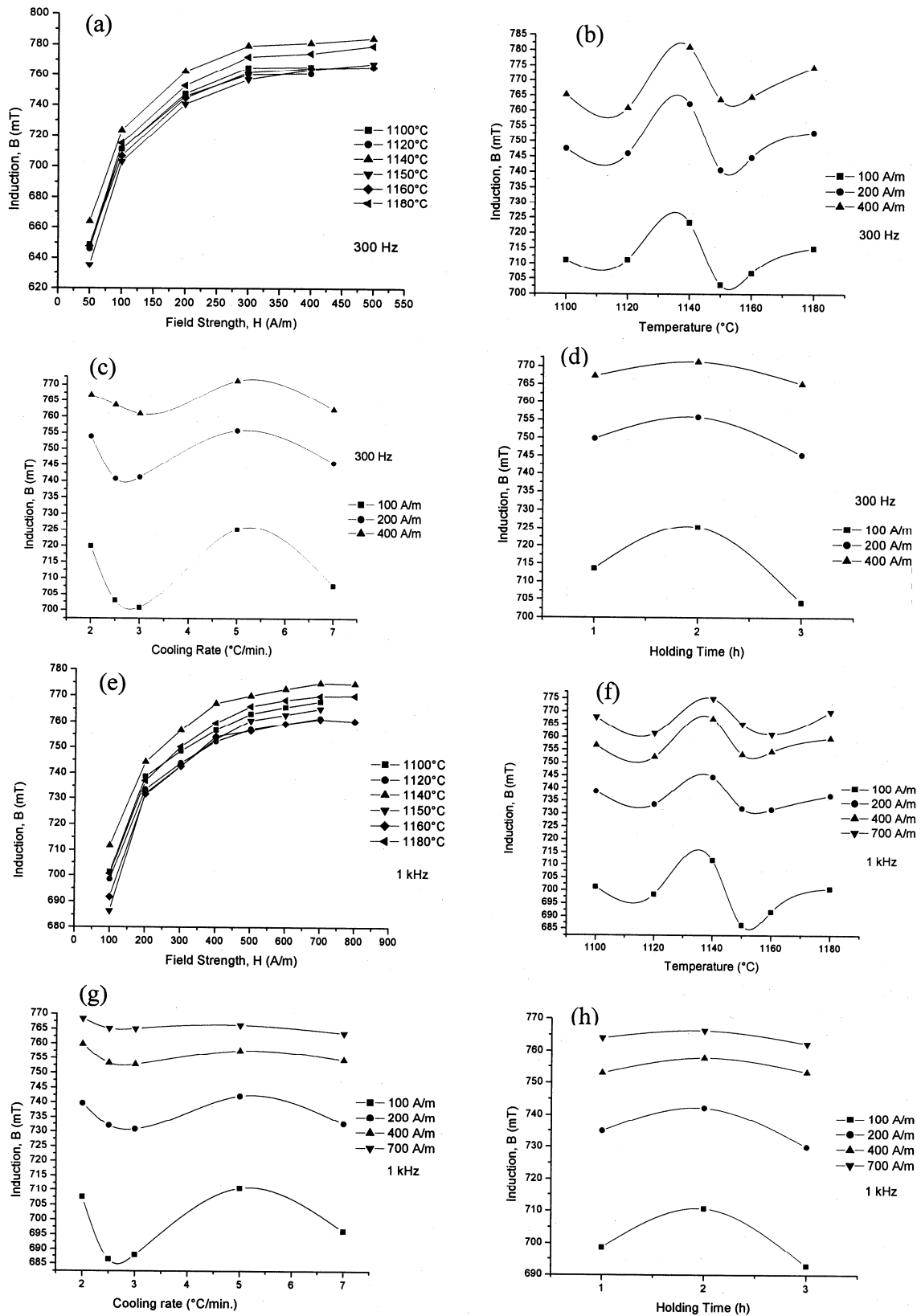


Fig. 3—Effect of field strength, annealing temperature, cooling rate and holding time on induction B at 300 Hz (a, b, c, d) and 1 kHz (e, f, g, h).

in field to about 400 A/m reduces the amplitude of this variation. We believe that it could be due to the misalignment in the domain growth at the lower field strengths that this variation is noticed but at higher field strength, the domain growth in various sub domains may result into the larger domain in order to achieve their saturation. Further increase in the field strength tends to change this curve into plateau indicating that a multi domain magnetic alloy is changed to a single domain magnetic material (as shown in Fig. 3a).

The variation of B as a function of holding time is shown in Fig. 3d. The result indicates a strong correlation between the holding time and field strength. For instance, at 100 A/m field strength, we see that B varies from 710 mT to 725 mT for a holding time of 2 h but decreases from 725 mT to 705 mT when the holding time increases, whereas at a field strength of 400 A/m the increase in B is not very appreciable for a holding time 1-2 h and also beyond 2 h the decrease is relatively very small at higher field strength that B becomes independent of holding time.

All the results convincingly signify that magnetic properties of permalloy can be controlled by these external parameters, however, ferromagnetic behaviour of the alloying elements (Ni and Fe) play significant role in higher induction.

Similarly, the behaviour of B at input frequency of 1 kHz as a function of field strength, annealing temperature, cooling rate and holding time is given in Fig. 3e-3h respectively. Similar behaviour of B is reported at 1 kHz frequency as compared to 300 Hz input frequency but the induction is higher at 300 Hz frequency as compared to 1 kHz frequency as given in Tables 3-5.

Remanence (B_r)

Similarly, the behaviour of remanence in Ni-rich samples corresponding to a function of field strength, annealing temperature, cooling rate and holding time at 300 Hz and 1 kHz frequency is given in Fig. 4a-4h respectively. We observed that at a input frequency of 300 Hz B_r increases from 465 mT to 475 mT where field strength increases from 50 A/m to 100 A/m thereafter decreases to 355 mT where the field strength increases to 500 A/m at 1150°C. Similar behaviour is depicted at another temperature as shown in Fig. 4a. The temperature dependence of B_r also shows an increase up to 1140°C, thereafter exponential decay at 1150°C and then B_r increases

with increase in temperature as shown in Fig. 4b. It is observed that the behaviour of remanence is opposite to the behaviour of B . We presume that it may be due to the field reversal, wherein the domains might tend to align opposite to initial direction. Thus at $H \approx 0$, B_r shows relative lower value as compared to B (Fig. 3b). These results are reported at a constant cooling rate of 2.5°C/min and holding time of 2 h. Also, B_r shows an interesting behaviour as a function of cooling rate as shown in Fig. 4c. The remanence decreases exponentially with increase in the cooling rate from 2 to 2.5°C/min at field strength of 100 A/m thereafter increases with increase in the cooling rate. The similar behaviour is also seen for other field strengths. The behaviour of B_r is also studied with respect to holding time at constant temperature 1150°C and at a cooling rate of 5°C/min as shown in Fig. 4d. It is noticed that values of B_r is almost constant at field strength of 200 A/m, whereas it decreases slightly from 425 mT to 420 mT at field strength of 400 A/m however, there is large variation is noticed at field strength of 100 A/m.

Fig. 4e-4h shows behaviour of B_r (at 1 kHz input frequency) as a function of field strength, annealing temperature, cooling rate and holding time. It is seen that the values of B_r are lower at 300 Hz as compared to 1 kHz as given in Tables 3-5.

Core loss (P_c)

An interesting behaviour of core loss as a function of field strength, annealing temperature, cooling rate and holding time at 300 Hz and 1 kHz frequency is given in Figs 5a-5h respectively. We observed that at 300 Hz, the core loss increases from 0.005 W to 0.019 W with increase in field strength from 50 A/m to 500 A/m for all studied temperatures (Fig. 5a). The temperature dependence of core loss shows stability at lower field strength of 100 A/m and shows variation at higher field strength for all studied temperatures as shown in Fig. 5b. This may be due to the localized heating effect of the material at such higher field strength. Similarly, the effect of cooling rate on core loss is shown in Fig. 5c. It is observed that the core loss varies where the cooling rate increases from 2 to 3°C/min, thereafter it becomes almost stable with further increase in the cooling rate up to 7°C/min at field strength of 100 A/m. But, the core loss shows variation at higher field strengths. However, the behaviour of core loss with respect to the holding time is stable (Fig. 5d). It is noticed that the holding time

Table 3—Effect of annealing temperature on magnetic properties of permalloy

Annealing temperature (°C)	B (mT)	B_r (mT)	Core loss (W)	H_c (A/m) (300 Hz)	Peak permeability
1100	711.13	505.62	0.008	36.40	5581
1120	711.25	507.93	0.008	35.85	5577
1140	723.44	509	0.008	35.79	5796
1150	703	475.02	0.008	35.12	5525
1160	706.73	501.96	0.008	35.55	5640
1180	714.99	514.94	0.009	36.37	5628
(1 kHz)					
1100	701.33	559.81	0.048	63.29	5483
1120	698.52	555.65	0.047	62.82	5458
1140	711.7	566.96	0.049	63.79	5565
1150	686.4	529.47	0.045	60.94	5500
1160	691.73	552.62	0.046	61.55	5430
1180	700.67	563.52	0.048	62.35	5371

Table 4—Effect of holding time on magnetic properties of permalloy

Holding time (h)	B (mT)	B_r (mT)	Core loss (W)	H_c (A/m) (300 Hz)	Peak permeability
1	713.67	496.04	0.008	36.18	5617
2	725.24	504.7	0.008	36.08	5739
3	704.02	491.21	0.008	35.41	5541
(1 kHz)					
1	698.67	548.17	0.047	62.19	5432
2	710.63	561.27	0.048	62.75	5560
3	692.74	545.66	0.046	61.34	5346

Table 5—Effect of cooling rate on magnetic properties of permalloy

Cooling rate (°C/min.)	B (mT)	B_r (mT)	Core loss (W)	H_c (A/m) (300 Hz)	Peak permeability
2.0	719.93	518.3	0.009	36.88	5708
2.5	703	475.02	0.008	35.12	5525
3.0	700.86	489.33	0.008	35.54	5512
5.0	725.24	504.7	0.008	36.08	5739
7.0	707.71	478.34	0.008	34.63	5583
(1 kHz)					
2.0	707.61	569.68	0.049	63.50	5587
2.5	686.4	529.47	0.045	60.94	5500
3.0	687.93	535.25	0.046	61.50	5367
5.0	710.63	561.27	0.048	62.75	5560
7.0	696.22	539.02	0.046	61.08	5422

does not have significant effect on the core loss at input frequency of 300 Hz. However, the behaviour of core loss is more stable at higher input frequency 1 kHz as a function of field strength, annealing temperature, cooling rate and holding time as shown in Figs 5e-5h respectively. But, the values of core loss

are higher at 1 kHz frequency as compared to 300 Hz frequency as given in Tables 3-5.

Coercivity (H_c)

An interesting behaviour of annealing temperature on coercivity at 300 Hz and 1 kHz frequency is shown in Figs 6a and 6b respectively. At higher field strengths,

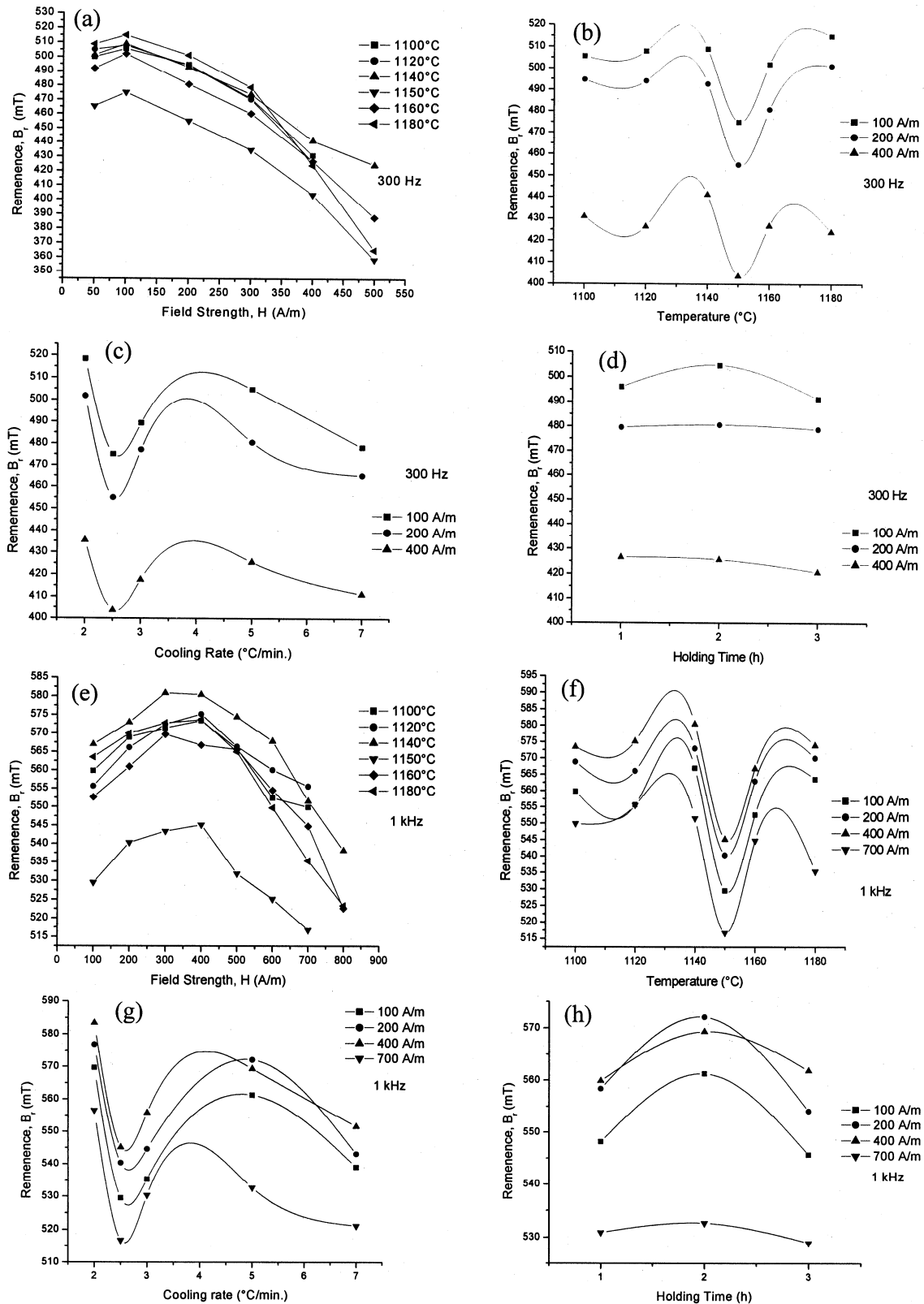


Fig. 4—Effect of field strength, annealing temperature, cooling rate and holding time on remanence B_r at 300 Hz (a, b, c, d) and 1 kHz (e, f, g, h).

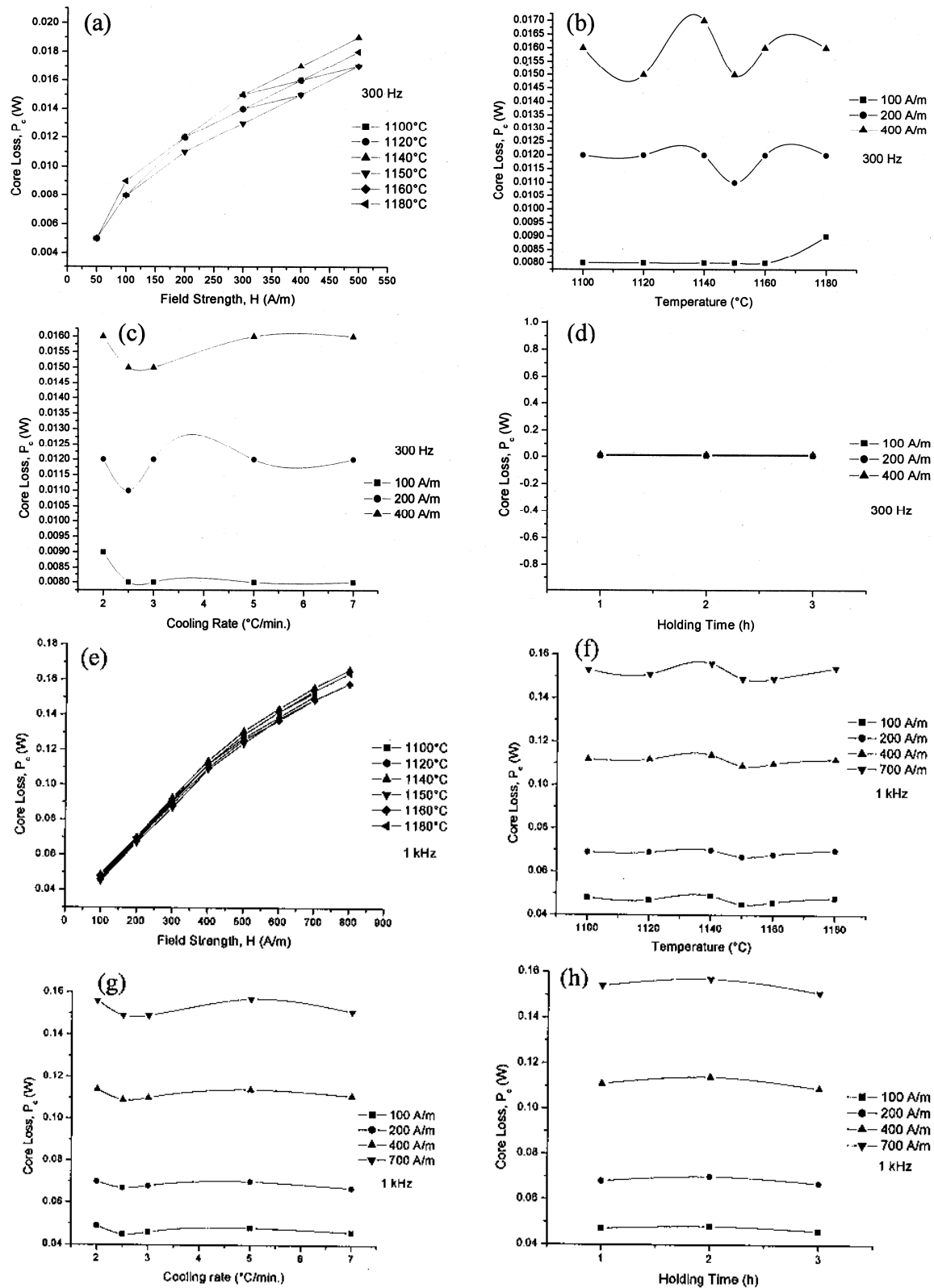


Fig. 5—Effect of field strength, annealing temperature, cooling rate and holding time on core loss at 300 Hz (a, b, c, d) and 1 kHz (e, f, g, h).

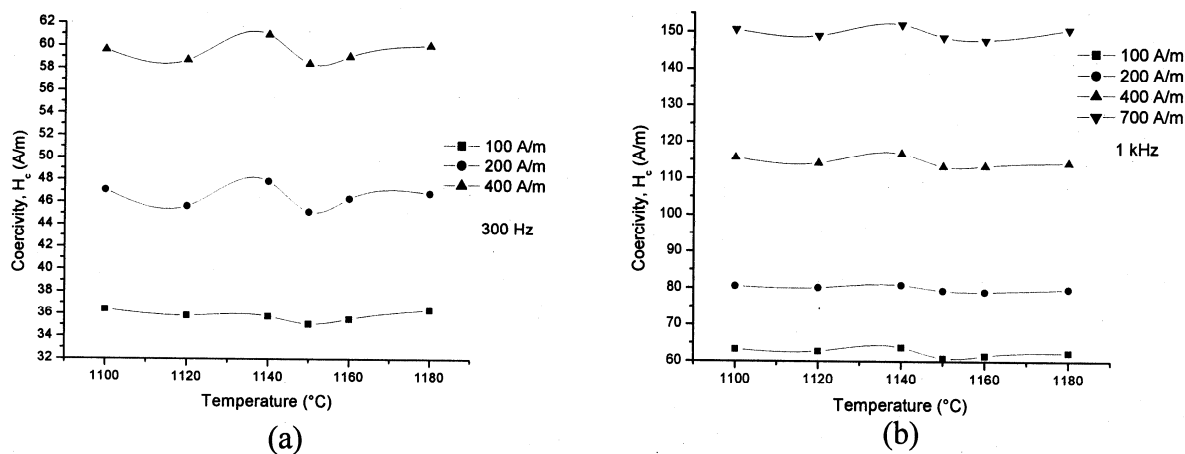


Fig. 6—Effect of annealing temperature on coercivity H_c at (a) 300 Hz and (b) 1 kHz.

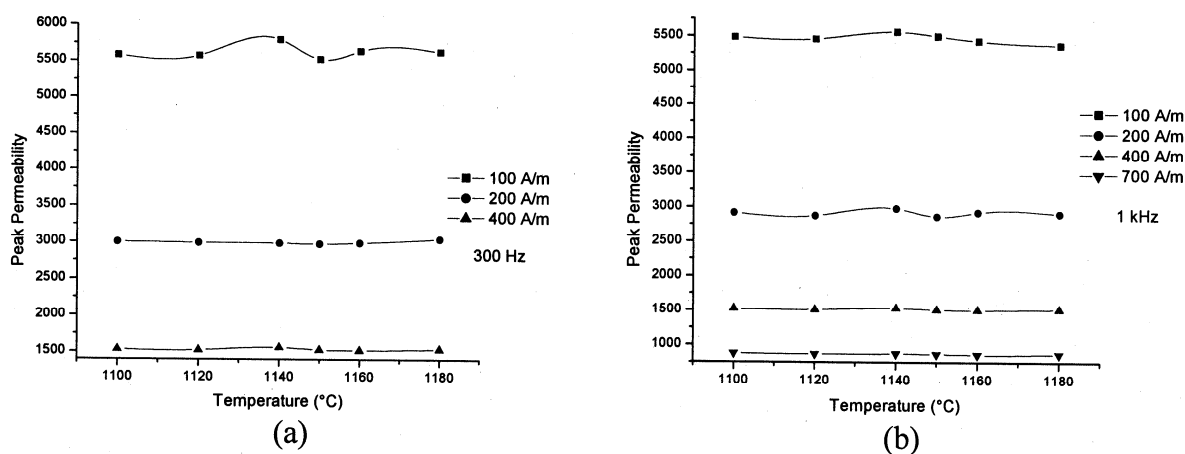


Fig. 7—Effect of annealing temperature on peak permeability at (a) 300 Hz and (b) 1 kHz.

i.e., 200 A/m and 400 A/m, it is observed that the coercivity shows variations in the intermediate temperature range 1120–1160°C where- as at 100 A/m the coercivity remains nearly constant indicating the stable performance of the device at this field (Fig. 6a). However, the behaviour of H_c is more stable at higher input frequency (i.e. 1 kHz) as a function of annealing temperature as shown in Fig. 6b.

Peak permeability

The behaviour of peak permeability as a function of annealing temperature at 300 Hz and 1 kHz frequency is given in Figs 7a and 7b respectively. The temperature dependence of peak permeability shows little variation at lower field strength of 100 A/m and at input frequency of 300 Hz but shows stability at higher field strengths, i.e., 200 A/m and 400 A/m as shown in Fig. 7a. Similar behaviour of peak permeability is reported at 1 kHz frequency as

compared to 300 Hz input frequency (Fig. 7b). The variation of coercivity and peak permeability as a function of various material parameters is given in Tables 3-5.

Conclusions

The magnetic characteristics of the 79.90 Ni-rich permalloy have been investigated under different conditions of annealing temperature, holding time and cooling rate. The experimental results indicate that these permalloys show better and improved magnetic properties at 300 Hz over 1 kHz frequency and at lower field strength indicate their better use at these frequencies.

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