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Round Robin Test Project in Universal Network for Magnetic NDE

Standardization of Magnetic NDE By Seiki Takahashi

ここに示す本論は2006年9月Pragueの国際ネットワークの会議で決定したラウンドロビン試験の結果を纏めたものである。ラウンドロビン試験の目的は磁気利用非破壊評価の基準化である。2007年9月Cardiff及び、2008年9月Budapestの会議で基準化の明確な方針を打ち出すことができなかった。筆者がラウンドロビン試験の結果を基に基準化の指針を示したのが本論である。この本論は第2回目のラウンドロビン試験がこの中で述べた指針に基づいて行われることを願い2008年11月にUNMNDEの全てのメンバーに送ったものである。

2-1 Participants and their measuring methods in Round Robin Test

Group	Country	sample		Repo rt	Measuring data		Experimental details
		#1	#2				
S. Takahashi	Japan	yes	yes	yes	WF0	Coefficients of power law relations between minor loop parameters	Analysis of set of quasistatic minor hysteresis loops f = 0.05 Hz (frame, Ring, plate), f = 0.5 Hz (Charpy)
					WR0		
					Hc0		
A. Moses	UK	yes	yes	yes	Vrms	RMS of BH signal	Magnetic Barkhausen Noise f = 50Hz Flux Density(steel): 0.1T (Charpy), 1T (frame), 0.02T (Plate) Flux Density(FeCu alloy) : 0.1T (Charpy B), 0.08T (Charpy C), 0.7T (Rings) Double coil method
					W	Power loss	Power Loss f = 50Hz Flux Density(steel): 0.1T (Charpy), 1T (frame), 0.02T (Plate) Flux Density(FeCu alloy) : 0.1T (Charpy B), 0.08T (Charpy C), 0.7T (Rings)
L. Dupre	Belgium	yes	yes	yes	Hc	Coercive field of saturation loop	Magnetic Hysteresis Cold rolled steel: * freq = 0.05 Hz (Charpy); 0.05 Hz (frame). * max field amplitude: 4 kA/m (Charpy); 5 kA/m (frame). Thermally aged Fe-Cu alloys: * f = 0.05 Hz (Charpy); 0.1 Hz (ring). * max field amplitude: 3 kA/m (Charpy B); 4 kA/m (Charpy C); 5 kA/m (ring)
					Mr	Remanent induction of saturation loop	
					W	Hysteresis loss of saturation loop	
					μ_{max}	Maximum of relative permeability along virgin curve	
					Qm	Peak value of distribution of local interaction field (Preisach analysis)	
					Qc	Peak value of distribution of local coercive field (Preisach analysis)	
G. Dobmann	Germany	yes	yes	yes	Hco	Coercivity derived from upper harmonics analysis	Upper Harmonics Cold rolled steel: $f_E = 100$ Hz, $H_{max} = 16$ A/cm Fe-Cu alloy: $f_E = 50$ Hz, $H_{max} = 5$ A/cm
					K	Distortion coefficient derived from upper harmonics analysis	
					Hcm	Coercivity derived from BHN analysis	Magnetic Barkhausen Noise Cold rolled steel: $f_E = 100$ Hz, $H_{max} = 16$ A/cm. bandpass-filtered to a range of 2-24 kHz. Fe-Cu alloy: $f_E = 50$ Hz, $H_{max} = 5$ A/cm. bandpass-filtered to a range of 4-24 kHz.
					DH25%	Half width of BHN curve at 25% from the maximum amplitude	
					Vmr	BHN amplitude in remanence area	

					Vmax	Maximum BHN amplitude	
B. Augustyniak	Poland	yes	yes	yes	Int-Ua	Integral of rms envelope of MAE	Magnetoacoustic emission (MAE) f = 1 Hz
					Int-Uan	Integral of pulse count of MAE	
					Int-Ub	Integral of rms envelope of BHN	Magnetic Barkhausen Noise f = 1 Hz, solenoid as source of field, pick-up coil
					Int-Ubn	Integral of pulse count of BHN	
J. Bydzovsky	Slovak	yes	yes	yes	μ	Differential permeability of MAT	Magnetic Adaptive Testing Cold rolled steel: (ha, hb) = (-300, 1800) in A/m unit Fe-Cu alloys: (ha, hb) = (560, 600) in A/m unit (Br1-Br4) = (-40, 80) in A/m unit (Cr1-Cr4)
					Vrms	RMS of BH signal	Magnetic Barkhausen Noise Fe-Cu alloy: f = 0.3 Hz, I _{max} = 1.5 A
I. Tomas	Czech	yes	yes	yes	Hc	Coercive field	Major hysteresis loop f = 0.05 Hz, H _{max} ~ 4 kA/m
					Mr	Remanent induction	
					μ_{max}	Maximum permeability	
					μ	Permeability of magnetic circuit	Magnetic Adaptive Testing F: magnetizing field, IF: magnetizing current, A: field amplitude, IA: current amplitude (1) Af1-Af5: dF/dt=704A/m/s 1/ μ : A=250A/m, F=200A/m; 1/ μ ': A=250A/m, F=125A/m (2) A1-A5 : dIF/dt=93.8mA/s along rolling direction; sample face without V-notch 1/ μ : IA=50mA, IF=20mA; 1/ μ ': IA=50mA, IF=0mA (3) A1-A5 : dIF/dt=94.1mA/s normal to rolling direction μ : IA=750mA, IF=600mA; μ ': IA=1100mA, IF=850mA (4) Br1-Br4: dF/dt=242.7A/m/s μ : A=100A/m, F=20A/m (5) Cr1-Cr4: dF/dt=121A/m/s μ : A=100A/m, F=70A/m; μ ': A=100A/m, F=50A/m (6) B1-B4 : dIF/dt=94.0mA/s along rolling direction; sample face without V-notch 1/ μ : IA=150mA, IF=130mA; 1/ μ ': IA=150mA, IF=90mA (7) C1-C4 : dIF/dt=94.0mA/s along rolling direction; sample face without V-notch 1/ μ : IA=200mA, IF=160mA; 1/ μ ': IA=200mA, IF=100mA
μ'	Derivative of permeability of magnetic circuit ($\mu' = d\mu/dIF$)						

G. Vertesy	Hungary	yes	yes	yes	μ	Permeability (optimized Magnetic Adaptive Testing descriptors)	Magnetic Adaptive Testing
F. Gillemot	Hungary	yes	yes	yes	Vexc_ip	Excitation at inflection point	Magnetic Barkhausen Noise Excitation frequency 10 Hz (sine wave)
					V_ip	MBN value at inflection point	
					V_sat	MBN value at saturation	
X. Kleber	France	yes	yes	yes	Vmax	Maximum amplitude	Magnetic Barkhausen Noise fexc = 55 Hz, Analysis: 10kHz ~ 5MHz
					PP	Peak position	
H. Hauser	Austria	yes	yes	yes	Hc	Cercivity	Major hysteresis loop Excitation frequency 0.01 Hz (triangular wave signal) Cold rolled steel: $H_{max} = 4$ kA/m Fe-Cu alloy: $H_{max} = 2.2$ kA/m
					Mr	Remanence	
F. Fiorillo	Italy	yes	yes	yet	Mr	Remanence	Magnetic hysteresis loop The measurements are performed at the frequency $f = 0.1$ Hz and the peak polarization value $J_p = 1.2$ T
					Hc	Coercivity	
					μ	Permeability at $J_p = 1.2$ T (μ/μ_0)	
					W	Energy Loss	
					a	Rayleigh constant	Rayleigh constant measurement ($J = aH + bH^2$) Measuring frequency $f = 10$ Hz
					b	Rayleigh constant	
E. Hristoforou	Greece	yes	yes	partial	μ_{max}	Maximum permeability	Magnetic hysteresis loop The measurements are performed at low fields. The data are obtained in V-I unit.
					Hc	Coercivity	
M. Pumarega	Argentina	yes	yes	yes	Vmax	Maximum peak to peak amplitude	Magnetic Barkhausen Noise traingular wave, excitaion frequency 1Hz and voltage 30V
					Vmax50	Vmax of 50 average	
S. Takahashi	Japan	yes	yes				REMEASUREMENTS: to check whether sample properties change in the course of round robin tests
D. Park	Korea	yes	yes	Patial	Vrms	RMS of BH signal	Magnetic Barkhausen Noise No information
L. Li	China	yes	yes	yes	Vrms	RMS of BH signal	Magnetic Barkhausen Noise Voltage=40V, frequency=100Hz, 5 times testing

As of 5th September 2007, two sets of samples(#1, #2) are circulated.

#1: A1-A5, Af1-Af5, B1-B4, Bb1, Br1-Br4, C1-C4, Cr1-Cr4 in Korea

#2: Ap1-Ap5 in Athens

Future plan

group	country	institute	period	method
A. Mitra	India	NML	5 weeks	Hysteresis, BHN
T. Jayakumar	India	IGCAR	6 weeks	Hysteresis, BHN
G.Y. Tian	UK	Univ. of Newcastle	2 or 3 weeks	MBN, ABN
C. Lo	USA	Iowa State Univ.	3 weeks	AC hysteresis, BHN
F. Landgraf	Brazil	Univ. of San Paulo		
E. Hristoforou	Greece	1 or 2 weeks for additional measurements		

2-2 Sample shape and size

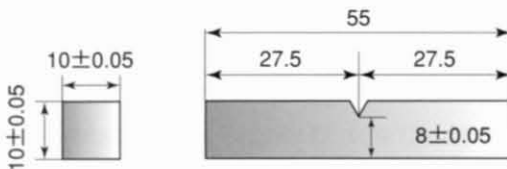
In Project 3 we make the standardization of magnetic properties connected with degradation in steels. We shall start the round-robin test in which each member practices his own measurements by use of the same samples. We compare all the measuring results during the Prague meeting in September 2006. We can offer two kinds of samples; A. cold-rolled steel samples and B. thermally aged Fe-Cu alloys.

A. Cold-rolled samples

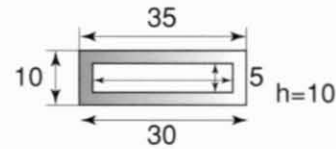
We shall prepare 5 cold-rolled samples of $\epsilon = 0\%$, 5% , 10% , 20% and 40% . The dislocation density was obtained by TEM and it is from 10^9 to 10^{10}cm^{-2} . Charpy impact test and Vickers hardness test have been done on such samples.

You can use these samples in your measurement. The samples shapes and sizes are the following;

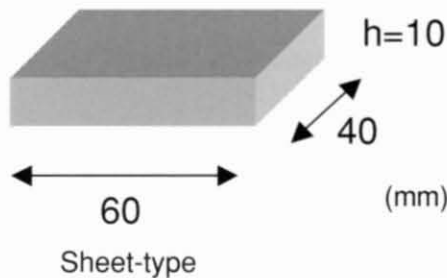
	C	Si	Mn	Fe
wt. %	0.15~0.20	0.15~0.35	0.30~0.60	bal.



Full-size Charpy specimen



Frame-type
with magnetizing and detecting coils

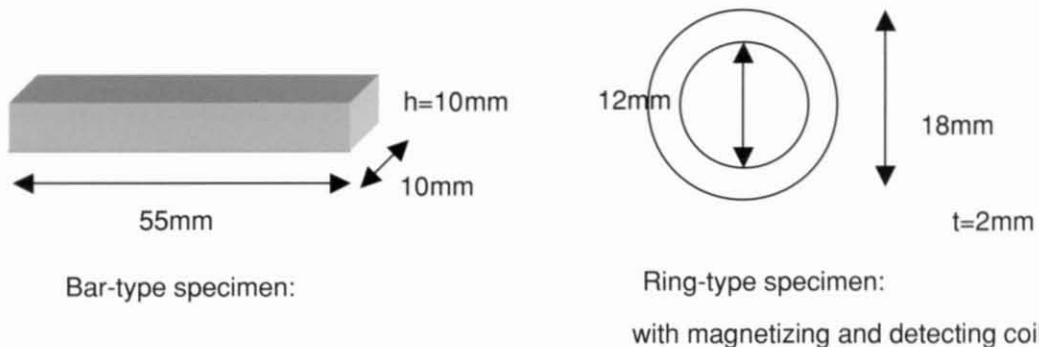


Sheet-type

B. Thermally aged samples

Thermally aged Fe-0.98 wt. % Cu alloys were prepared for modeling neutron irradiation in nuclear reactor pressure vessels. By aging the samples at 500°C, Cu precipitates are formed in the Fe metal matrix. There will be 5 samples with different ageing time in the series. Vickers hardness and coercive force were measured on such samples and confirmed the material was substantially changed.

The samples shapes and sizes are the following;



Kindly answer this announcement and tell us if you are interested in the round robin test. We have to make a time-schedule and to know your circumstances.

Will you join the round robin test? 1)Yes 2)No

Which kinds of samples are you interested in?

- 1) We are interested in the cold-rolled samples.
- 2) We are interested in the thermally aged Fe-Cu samples.
- 3) We are interested in both the series of samples.

What are your measuring methods?

What is your measuring schedule?

- 1) We can do the measurement any time.
- 2) The measurement is possible only in the following time-period:

How long time do you need for your measurement?

Our measurement will take:

After obtaining answers from all the members of the Universal Network (but latest on December 31th 2005) the timetable and the order of the UN members in the round robin tests according to the above data will be determined and circulated to all the members. Sending of the first samples to the first laboratory according to the timetable can be expected in mid March 2006.

2-3. Standardization of magnetic NDE

We started Round Robin Test among UNMNDE members for the standardization of magnetic NDE and have valuable data now. We have consistent results and inconsistent ones. I believe the first Round Robin Test is successful and fruitful. The second Round Robin Test will soon start. We have to give an evaluation to the previous results to make the second Round Robin Test more successful and fruitful.

We discussed our results in the UNMNDE meeting of Budapest. I summarized our results according to your opinions and have made a report on Round Robin Test. My evaluation is not necessarily correct in this report. If you have any argument against my opinion, please tell me your argument. The most important item is shown in the last section XII "Proposal". I wish that this report triggers animated discussion over the standardization of magnetic NDE.

2-3-1. Round Robin Test and evaluation

Round Robin Test helps us to know several issues for the standardization of magnetic NDE. Some properties obtained from hysteresis loops show a good agreement with each other in the closed samples (see Fig.1). In opened samples, however, we could not have good agreement in our results even in coercive force (see Fig. 2). The results by Barkhausen noise effect showed a large disagreement among each group (see Fig.3).

The disagreement is an unexpected result for us, since these magnetic properties are caused of the interaction between magnetic domain walls and the lattice defects such as dislocations. As Prof. Kronmüller said, that magnetic domain wall is a sensor in the magnetic NDE method. All the properties should change consistently with lattice defects. But our data do not necessarily agree consistently with each other.

It is difficult to say which results are correct or wrong for the different results. But we have to judge our results, for the standardization of magnetic technique. It would be the best way to decide which is correct or wrong that each group evaluates his results and techniques comparing with the others.

As Stuttgart group in Max-Planck Institute says, coercive force, the initial susceptibility and Rayleigh constant has a simple function of the dislocation density ρ .

$$H_c \propto \rho^{1/2},$$

$$\chi_0 \propto \rho^{-1/2},$$

$$\alpha \propto \rho^{-1},$$

where

$$B = \chi_0 H + \alpha H^2.$$

The relation represents the pinning effect of dislocations. We have data supporting the theoretical relation.

2-3-2. Coercive force as a standard property

Coercive force is a reliable magnetic property experimentally and theoretically and possible to be obtained independent of the demagnetization effect. The reliable measurement for coercive force is possible for the closed samples as well as the open samples. It was measured in the Round Robin Test by several groups. Rayleigh constants and hysteresis loss would be standard properties too. And Rayleigh constants and hysteresis loss should be measured by many groups in the second Round Robin Test and the measuring technique to them can be evaluated by the comparison of each results.

The values of coercive force are nearly the same in the closed samples (see Fig. 4) but deferent in open samples (see Figs. 5 and 6). The deference is caused of the measuring technique. The measuring technique should be improved in the open samples comparing with the closed ones. Coercive force is not disrupted by the demagnetization effect and the values of open samples should agree with the closed ones.

2-3-3. Coercive force and the other properties

We have the other magnetic properties but for “coercive force”. The other magnetic properties should agree with “coercive force” in their trend fundamentally. Absolute values are difficult to be obtained in some properties. But the trend of the relative ones should agree with “coercive force”. In the properties obtained from hysteresis loops, the values in closed samples are reliable compared with opened samples. If some property disagrees with “coercive force”, we should explain the disagreement from the physical point of view.

2-3-4. Remanence

“*Remanence*” is a magnetic structure sensitive property and shows a consistent relation with coercive force (see Figs. 7 and 8). The values obtained by us agree with each other in the closed samples (see Fig. 7). The relation between “*remanence*” and dislocations is not simple and its value is decided by the other factors, such as the demagnetization effect. “*Remanence*” is not an appropriate property to get the information of lattice defects.

2-3-5. Direction of standardization

We have many magnetic structure sensitive properties. Some of them are not appropriate to NDE from two causes mainly. One is caused of the fundamental reason such as “remanence”. The other is caused of the measuring technique. The later would be conquered by the improvement of our technique. We should assign the reason for the disagreement of our data, due to the first reason or the second.

2-3-6. Samples of Round Robin Test.

1. Cold-rolled low carbon steel (picture frame samples, Charpy samples and plate samples)
2. Aging at 773 K in Cu-Fe model alloy (ring samples, Charpy samples and plate samples).
 - a) without pre-strain
 - b) with pre-strain $\epsilon = 10\%$

The change of magnetic properties is very small in the aging samples without pre-strain. The change is clear in cold-rolled steel samples. The microstructure of materials is the same fundamentally in frame samples, ring samples, Charpy samples and plate samples, though small difference exists among them.

2-3-7. Minor loop properties

Minor loop properties are sensitive to the microstructure of materials compared with the major loop properties. Their relation with lattice defects, however, has not been studied fully from the theoretical and experimental point of view. The minor loop properties depend on the magnetic amplitude, though some of them are independent of the amplitude. How to conquer the latter shortcoming would be the present problem.

We have an agreement among the minor loop properties obtained by each group (see Figs. 9 and 10), though a few disagreements exist in the heavily deformed samples. The trend and the high sensitivity in heavily deformed samples should be explained in terms of physics.

MAT method has the same characteristic as the minor loop methods. MAT is sensitive to the microstructure of materials compared with the minor-loop properties by Takahashi group and Dupre group as well as the major loop properties. But MAT method has two shortcomings.

1. Their relation with lattice defects has not been studied theoretically.
2. MAT properties depend on the magnetic amplitude and magnetic field.

The first shortcomings would be conquered by the theoretical study. The second shortcoming reveals in the sensitivity that depends considerably on the shape of samples (see Figs. 11 to 14). The different sensitivity in the sample shape would become difficulty of NDE against the actual objects with various shapes. The sensitivity would be different in the position of an object.

The result of Vertesy is the same as that of Bydzovsky in the trend of $1/\mu$ but their results are different from that of Tomas (see Figs. 11, 12 and 13).

2-3-8. Summary

@ Summary 1 MAT

1. MAT properties depend on the amplitude of minor-loops as well as the magnetic field. Their sensitivity depends on the shape of samples strongly (see Figs. 10 and 14).
2. MAT properties are much more sensitive than the others, 5 to 20 times of coercive force.
3. MAT properties have a consistent relation with coercive force but do not have a linear relation

with coercive force (see Figs.10 to 12).

@ Summery 2 Takahashi method

1. The minor-loop properties are independent of the amplitude of minor loops as well as the applied field.
2. The properties have a good relation with coercive force and are more sensitive to lattice defects than coercive force (see Figs. 8).
3. The sensitivity of the properties in closed samples is nearly the same as those of opened samples (see Figs. 8 and 15).

@ Summery 3 The other minor-loop properties

1. The properties depend on the amplitude of minor-loops.
2. The minor-loop properties are more sensitive to lattice defects than coercive force (see Fig. 10).
3. The property $1/Q_m$ does not necessarily have the linear relation with coercive force (see Figs. 9 and 10).

@ Summery 4 Traditional

1. The traditional properties Rayleigh constant, hysteresis loss and initial susceptibility were measured by only one or two groups.
2. The traditional properties except for remanence have a simple relation or a linear relation with coercive force.
3. Rayleigh constant has a higher sensitivity than coercive force.
4. The measuring technique of them is more difficult than that of coercive force.

2-3-9. Barkhausen noise method

BHN methods are convenient to the measurement independent of the shape of objects. BHN effect has been studied theoretically and experimentally and the method has been expected to be NDE for degradation before crack initiation.

The data of Barkhausen noise effect depend on the each measurement method (see Figs. 16 to 23) though the data are consistent with “*coercive force*” in their trend. The reasonable conditions for measurement should be found comparing with coercive force Almost data have a linear relation with “*coercive force*” (see Figs. 16, 20 21 and 23). The disagreement with “*coercive force*” appears in heavily deformed samples (see Figs. 16 and 17). The decrease of V_{rms} in the hard deformation would be caused of the measuring condition.

The reasonable conditions for measurement to such as the frequency and the amplitude of the applied field should be found comparing with “*coercive force*” . We have to conquer this item for the standardization of BHN methods.

2-3-10. Evaluation of hysteresis methods to each group method

We have our own methods or special techniques that have been developed by ourselves. Some of them were discovered recently and have not been necessarily examined from the wide point of view. I have pointed out my viewpoints according to their data. My evaluation is not necessarily correct. If you have any argument against my opinion, please tell me your argument.

a) Takahashi group

Minor-loop properties are independent of the applied magnetic field as well as the amplitude of minor loops. Their change against dislocations and Cu precipitates agrees with that of coercive force (see Figs. 25, 26 and 27). Their sensitivity is higher than that of coercive force. The measuring technique is poor comparing with the other groups; especially in open samples (see Figs. 5 and 6). The poor technique appears in the Charpy samples aged without pre-strain in which the relations appears in the second quadrant (see Fig. 28). The linear relation between minor-loop properties and coercive force is obtained in the thermally aged Fe-Cu opened samples (see Figs 29, 30 and 31).

b) Tomas group

1. The minor-loop properties show a consistent change against coercive force, except for Fe-Cu alloy aged thermally without pre-strain (see Figs. 35 and 36).
2. MAT properties are very sensitive to the microstructure of materials (see Figs.32 to 37). The properties depend on the applied field as well as the field amplitude. MAT values depend on the sample shape strongly.
3. The relation between MAT properties and coercive force appears in the second quadrant in the thermally aged Fe-Cu alloys (see Figs. 35, 36 and 37). This fact should be explained reasonably.
4. MAT properties have the non-linear relation with coercive force (see Figs. 32 and 37). We have to examine whether the non-linearity causes of the fundamental reason or of the measuring technique.

c) Dupre group

The properties, W , $1/\mu_{Max}$ and $1/Q_c$ change consistently with coercive force in the ring samples (see Figs.39 and 40).

These values in Charpy samples are nearly the same as that of ring samples (see Figs. 39 and 40). The measuring technique for opened samples is in the high level.

The minor-loop properties depend on the amplitude but their sensitivity does not show the remarkable difference in the samples. It would be caused of the fact that the minor-loop amplitude is enough compared with the major loop. The relation of coercive force and the other magnetic properties does not show the linearity in the Fe-Cu samples aged thermally without pre-strain (see Figs. 41 and 42). But the relation shows the nearly linear relations the Fe-Cu

samples with 10 % pre-strain (see Figs. 43 and 44).

d) Fiorillo group

The properties, W and $1/\mu$ show the linear relation with coercive force. Rayleigh constant $1/b$ is more sensitive than the other properties but do not show the linear relation with coercive force (see Fig. 45 a and 45 b). The non-linearity would be explained by the dislocation dependence of $1/b$ as shown in section I.

The relation of coercive force and the other magnetic properties does not show the linearity in the Fe-Cu samples aged thermally without pre-strain (see Fig. 46). But the relation shows the nearly linear relations the Fe-Cu samples with 10 % pre-strain (see Fig. 47).

e) Hauser group

The results of coercive force and remanence agree well with the other groups (see Figs. 4 and 7). Remanence is not appropriate for NDE of lattice defects as discussed in section IV (see Fig. 48).

f) Hristoforou group

The magnetic susceptibility obtained by a minor loop shows a consistent relation with coercive force, though it is non-linear relation (see Fig. 49). The non-linearity should be examined whether it is cause of the fundamental or of the technical one.

2-3-11. Evaluation of Barkhausen noise methods

The data of Barkhausen noise effect depend on the each measurement method and some data are not consistent with coercive force in their trend. Almost results show a linear relation with coercive force. Dobmann group (Figs. 22 and 23), Bydzovsky group (Figs. 19, 20 and 21) Luming group (Figs. 16 and 20), Augustyniak group (Fig. 16 and 21), Kleber group (Fig. 23) and Park (Figs. 20 and 21).

We have non-linear results with coercive force in the cold-rolled samples (see Figs. 16 and 17). BHN properties as well as coercive force change remarkably in the cold-rolled samples compared with the thermally aged samples. The reason of the non-linearity should be explained.

The sensitivity of BHN properties is not necessarily higher than that of coercive force, though the advantage of BHN measurement is better than that of coercive force.

Remark

The relation of coercive force and the magnetic properties does not show the linearity in the Fe-Cu samples aged thermally without pre-strain (see Figs. 28, 35, 36, 41, 42 and 46). The aging effect is very small in the samples without pre-strain and the measuring error would be contained in both coercive force and the other properties.

2-2-12. Proposal

1. The magnetic properties should be evaluated on the bases of “coercive force” obtained in the closed samples.
2. Absolute values are difficult to be obtained in general. But the trend of the relative ones should agree with “coercive force” obtained in the closed samples
3. If some property disagrees with “the coercive force” , we should examine the cause of disagreement, which cause it comes from.
4. The values in closed samples are much more reliable than in opened samples in the hysteresis methods. Our goal is NDE for the opened samples. The measuring technique for the opened samples should be improved comparing with the data of closed samples.
5. BHN measurement can be carried out in the opened samples. The BHN properties should be compared with “coercive force” obtained by the closed samples too.
6. The values of BHN should change consistently with “coercive force” . If they do not, the cause should be explained from the physical point of view.
7. The values of a BHN property as well as a hysteresis loop one that are measured by different groups should be the same ultimately.

ここに示すデータはラウンドロビン試験結果を私の提案した指針に基づいて解析したものである。用いられている物理量の記号の説明は” 2-1 Participants and their measuring method in round Robin Test”にある。磁氣的物理量が保磁力 H_c と線形の関係、又は単純な関係にあるか見て欲しい。例えば、残留磁化（レマネンス） M_r は保磁力 H_c と線形の関係にない（Figure 7、Figure 8参照）。

2-3-13 Data of Round Robin Test