THE PRODUCTION OF CALIBRATION STANDARDS FOR DELTA-FERRITE

Flávio A.S.A. Souza
Dorival G. Tecco

SUMMARY

The production of mixtures with controlled proportions of magnetic and non-magnetic powders is investigated as a means of arriving at calibration standards for delta-ferrite volume fractions in stainless steels. Initially, the effect of substrate chemical composition was assessed with a range of magnetic alloys having variable Fe content, using a ferritoscope. No variations were detected amongst alloys, so the subsequent study was narrowed to two magnetic powders only, one 100% pure Fe powder and another experimental 19%Cr-81%Fe alloy powder. Several equivalent volume fractions were prepared with each of the two powders separately, using a non-magnetic 19%Cr-11%Ni-70%Fe powder. The ferritoscope revealed itself as strongly insensitive to actual void fraction, the readings obtained with the loose powder mixtures matching the reference calibration scale. A procedure was devised to fabricate pressed samples from the powder mixtures. The readings yielded by those pressed samples were consistently weaker than the reference calibration scale, possible reasons and corrective measures being pointed out.

INTRODUCTION

The importance of the delta-ferrite fraction in austenitic stainless steels is well known in defining a range of factors such as susceptibility to cracking, properties and in-service behavior, Ref.(1). Several measurement methods were developed in the past, including magnetic balances, optical metallography, X-radiography, Mossbauer effect, electrochemical dissolution, etc., Refs. (2-5). Despite the number of methods for measurement, no consensus has yet been reached on a calibration method giving reliability over the full delta ferrite volume range from 0% to 100%.

This work describes a procedure and the preliminary data obtained in the production of calibration standards for delta-ferrite fraction using controlled powder mixtures. The principle behind the procedure consists in defining the proportions of magnetic and non-magnetic powders from previously estimated Equivalent Magnetic Volume Fractions (EMVFs), assuming full densification. Although extensible up to 100% EMVF, the present range was deliberatively limited to 30% EMVF in order to fit the existing instrument scales.

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EXPERIMENTAL

The experimental programme consisted in determining the departures in magnetic response from a fixed reference, as caused first by changes in chemical composition and then by varying proportions of magnetic and non-magnetic powders.

The influence of chemical composition was assessed comparatively by testing four different magnetic substrata, including

i. one wrought C-Mn steel substrate,
ii. one wrought 12%Cr-88%Fe alloy substrate,
iii. one experimental 40%Cr-4%Ni-56%Fe alloy cast in a 5 kg Vacuum Induction Melter (VIM) and
iv. one experimental 19%Cr-81%Fe alloy, also cast in a 5 kg VIM.

Two magnetic powders having different chemical compositions were used, one pure Fe powder and another 19%Cr-81%Fe powder generated from the same alloy mentioned in (iv) above. Only one non-magnetic powder having a 19%Cr-11%Ni-70%Fe composition was used throughout. Particle sizes and additional specification are given in Table 1.

Two groups of mixtures with varying EMVFs were prepared, according to Table 2. The individual volume fractions were computed prior to mixing from the individual specific masses, considering a fixed final mass of 6 g and assuming perfect compaction (i.e. zero nil void fraction). Weighing accuracy was 0.001 g. Mixing was obtained in an orbital mixer for a period not inferior to 2 hr.

The magnetic readings were restricted to the use of a ferritoscope only, the inclusion of other instruments being planned for the future. The instrument presently employed was a type C model supplied by Fisher Instrument (Germany) and meeting AWS standard A4.2-74, Ref.(2). Only one probe having a 4 mm electrode centerline spacing was employed throughout.

The experimental error was estimated as less than 5% for readings up to 30%. Resolution was better than 0.15%, 0.4% and 1.5% respectively for the scales of 3%, 8% and 30% available in the instrument.

POWDER COMPOSITION, Wt.%	CODE	PARTICLE SIZE, Micrometers
19%Cr-81%Fe	A	less than 40
100%Fe	B	less than 40
19%Cr-11%Ni-70%Fe	C	from 37 to 53

TABLE 1: CHARACTERISTICS OF THE POWDERS EMPLOYED IN THE SAMPLES

NOTES: (1) Powders A and B are magnetic.
(2) Powder C is fully non-magnetic.

MIXTURE	ESTIMATED VOL.FRACTIONS,% A	B	C
1	1	0	99
2	0	1	99
3	5	0	95
4	0	5	95
5	10	0	90
6	0	10	90
7	20	0	80
8	0	20	80
9	30	0	70
10	0	30	70

TABLE 2: EXPERIMENTAL MIXTURES OF MAGNETIC AND NON-MAGNETIC POWDERS

NOTES: (1) Key to powders and respective compositions in Table 1
(2) The Volume Fractions are estimated from individual specific masses, assuming nil void fraction

The reference magnetic calibration scale (hereafter denominated "reference calibration") was established according to the instructions provided by the instrument manufacturer, using a standard magnetic substrate and four plastic thickness gages (Table 3), which create an equivalent air gap between probe and substrate.

Both loose and pressed powders were tested in the experiments. All pressings were carried out immediately after mixing to prevent against loss of uniformity, with a 46200 kg load to final dimensions of 14 mm dia. by 5 mm thickness. No subsequent heat treatment was applied to avoid diffusion or oxidation. The pressed samples were then impregnated with a liquid resin under vacuum at room temperature. Readings were made in the controlled samples in three planes: each of the two round faces and the axial plane (axial plane being prepared through sample sectioning).

THICKNESS OF PLASTIC GAGE, mm	FULL SCATTER BAND READINGS, %
1.75	2.6-2.7
0.75	8.4-8.6
0.225	19.7-20.5
0.075	28.7-29.0

TABLE 3: REFERENCE CALIBRATION SCALE

RESULTS AND DISCUSSION

The reference calibration scale (Table 3) was reproduced with all four solid magnetic substrata and four plastic thickness gages, indicating no effect of chemical composition over the ranges and conditions investigated. Though quite convenient from an experimental point of view, it must be remembered that distinct trends may be expected with other methods (e.g. magnetic balances, Ref.(6)), so care must be exercised before extrapolations are attempted.

The readings obtained with the 19%Cr-81%Fe and 100%Fe magnetic powders both in the loose and in the pressed conditions again reproduced the reference calibration scale with all four plastic gages. That was rather unexpected, considering the widely different void volume fractions amongst samples, i.e.

i. nil volume fraction for the cast alloy,
ii. about 30% void volume fraction for the pressed powder (as determined from quantitative metallography) and
iii. a comparatively "very large" fraction with the loose powder.

The insensibility of the instrument to the presence of large void fractions was again reproduced with the controlled powder mixtures. As listed in Table 4, the readings coincided with the reference calibration scale with all magnetic proportions up to 30%. More data will be required before it can be decided whether that was caused by specific magnetic interactions between probe and powder or electronic biasing.

Table 5 shows that the pressed mixtures had a weakened magnetic response with respect to the reference calibration scale. It was also observed that the pressed samples using powder A gave weaker readings than those corresponding to powder B, with a detectable loss of linearity in the latter case. Although the action from void fraction may not be yet excluded, it was strongly indicated that the trends were associated to an insufficient sample diameter, albeit the small probe spacing. Larger samples are being produced and results are expected soon.

MIXTURE	FERRITOSCOPE READING (SEE NOTES)
1	1.3
2	1.0
3	5.5
4	5.5
5	9.9
6	10.0
7	19.1
8	20.0
9	28.5
10	29.5

TABLE 4: FERRITOSCOPE READINGS WITH THE CONTROLLED POWDERS IN THE LOOSE CONDITION

NOTES: (1) Key to powders and respective compositions according to Tables 1 and 2
(2) Measurements made with the probe dipped into the powder mixture

MIXTURE	FULL SCATTER BANDS IN FERRITOSCOPE READINGS, %	
	THE TWO ROUND FACES	THE AXIAL SECTION
1	0.8-1.0	ND
2	0.4	0.3-0.4
3	2.4-2.7	ND
4	1.5-1.9	1.5-2.0
5	4.4-4.5	ND
6	3.3-3.9	2.5-4.5
7	8.2-9.3	ND
8	10.6-12.5	10.0-12.5
9	11.0-13.5	-
10	16.0-23.7	17.0-25.0

TABLE 5: FULL SCATTER BANDS IN READINGS WITH THE PRESSED POWDER MIXTURES

NOTE: Void fraction was about 30% for all samples

CONCLUSIONS

The use of controlled powder mixtures is a potential route for the production of delta-ferrite calibration standards because it makes it possible to combine specific properties in each separate phase, while still maintaining an uniform dispersion.

Although valid in the present scope, the ferritoscope results were marked by some peculiarities and an extension is required to include other measurement methods. The ferritoscope apparent insensitivity to void fraction was noteworthy and needs to be re-assessed in greater detail. Despite the considerable care and planning in making the pressed samples, their diameter was apparently too small and will have to be increased.

Future investigation is also necessary to identify the influences of factors such as void densities (within more controlled limits), absolute and relative sizes of the magnetic and the non-magnetic particles and magnetic network continuity (as compared with a discrete distribution of isolated particles).

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