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ABSTRACT

The magnetic saturation method can be used to determine absolute delta ferrite content of an austenitic weld metal. This report describes an apparatus based on above method that has been developed at Reactor Research Centre. With this apparatus ferrite measurements can be carried out to an accuracy of $\pm$ 1% ferrite. The report also briefly reviews other methods of measuring absolute ferrite content. Results obtained with the apparatus developed at RRC are compared with results from metallographic method and ferritescope and from a similar equipment in another laboratory.

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1. INTRODUCTION

Most austenitic stainless steel weld metals are produced to contain small amounts of delta ferrite in the weld in order to prevent cracking and microfissuring. It is generally considered sufficient to produce weld metals with greater than 3% ferrite to control hot cracking [1]. However, in certain applications where fabricated stainless parts must operate at elevated temperature, say about 550°C, the presence of delta ferrite could cause sigma phase formation resulting in appreciable losses in impact strength and corrosion resistance. In such cases, it is essential to impose a limit on the maximum delta ferrite content of the weld metal. For the Fast Breeder Test Reactor under construction at Kalpakkam, ferrite requirement in the stainless weld metal has been specified in the range of 2.5 - 5.5%.

This report discusses the magnetic saturation method which can accurately measure absolute ferrite content of the weld metal. An apparatus based on this method developed at the Reactor Research Centre is described. The report also briefly reviews some of the other methods for determining absolute ferrite content.

2. MEASUREMENT OF ABSOLUTE FERRITE CONTENT OF THE WELD METAL

Certain properties such as high temperature impact strength and ductility of austenitic stainless steels have been found

to largely depend on the overall ferrite content of the weld metal[2]. This is of particular importance to components of a Fast Reactor which continuously operate at high temperature (in the neighbourhood of 550°C). There is a need, therefore, to determine and limit the absolute ferrite content of the weld.

It has been, however, found that ferrite distribution in a weld metal is quite non-uniform. In a 2.7 mm GTA weld, for example, ferrite was observed to vary from less than 0.5% at the root to more than 7.5% at the face of the weld[3]. Similarly ferrite content at the fusion zone with base metal is small and increases towards the centre of the weld. Because of this and because the commercial magnetic instruments generally used (e.g. Magne gage, Ferrite scope etc.,) read local ferrite content and also depend on the size shape and orientation of ferrite particles, it is practically impossible to determine absolute ferrite content with such instruments. Some of the methods of estimating or measuring absolute ferrite content of a weld metal are briefly discussed in the following paragraphs. This is followed by a detailed description and discussion of Magnetic Saturation Method.

2.1. Constitution Diagrams for Determining Ferrite

The well-known Schaeffler diagram relates the chemical composition, by means of a chromium equivalent versus a nickel equivalent, to the expected ferrite content in the weld metal. It was based on quantitative metallographic measurements and some unspecified magnetic methods for ferrite determination[4]. The accuracy of this method according to Schaeffler himself, is $\pm$ 4 per cent ferrite especially in the lower range upto 5 per cent ferrite[5].

De Long published an adapted diagram mainly to emphasize the role of nitrogen [6]. The measurements were made by Magne gage with the calibration curve checked by weld metal samples in which the ferrite content had been primarily determined by quantitative metallography. The accuracy of this diagram has been claimed to be ± 3 per cent ferrite [7]. According to Ratz and Gunia [8] the ferrite percentage can be as high as 2 to 3 times that predicted by the constitution diagrams, for ferrite contents less than 10%.

2.2. Quantitative Metallography

On the assumption that the volumetric proportions of a three dimensional test specimen can be considered equal to proportions viewed on a random two dimensional plane, the ferrite content can be measured by scrutinizing a plane.

Metallographic measurements are helpful, but they are difficult to perform accurately. The fineness of ferrite particles and inherently different corrosion responses of ferrite of varying analyses to different etching media, make an optimum etch rather difficult to establish and an accurate result difficult to obtain. Moreover, ferrite is quite variable locally within the bead and from bead to bead [7].

According to Ratz and Gunia [8] the precision of this method is limited to ± 3% ferrite upto 10% ferrite and ± 6% ferrite from 10 to 24%.

According to Mudde, [4] the quantitative metallographic methods can only give values on an arbitrary scale and these values make sense only when given with a reference to the method used.

2.3. Mossbauer Method[9]

This method measures the relative amounts of austenite and ferrite phases on the basis of their magnetic properties, but in a way which is relatively independent of the shape, size and orientation of ferrite particles.

In this technique, the sample containing ferrite is irradiated by means of 14.4 KeV gamma rays emitted by a Co^{57} radioactive source. The scattered or transmitted radiations from the sample are detected by appropriately placed counters. The different phases in a metal give well resolved Mossbauer spectra (as observed on an oscilloscope connected to the spectrometer) and the relative areas of the spectral patterns provide an indication of the amount of each phase present. It is claimed that this method measures absolute ferrite content. This, is however, true for only a small portion of the sample that can be probed. (Sample thickness of only 100 microns can be normally probed). Considering the variations in ferrite content in the thickness of a weld sample, it is felt that this method cannot give overall ferrite content of a weld metal. Moreover, it is reported that the precision of the Mossbauer method is less than that of the magnetic saturation method[9].

2.4. Magnetic Saturation Method

This is based on the following principle: The intensity of magnetisation at saturation of a sample of given composition is proportional to the quantity of magnetic phase (ferrite in this case) in the sample.

In this method, a sample of specified dimensions from a weld metal pad containing ferrite is magnetized at different

field strengths varying between 1700 and 2500 Oe and the intensity of magnetization is measured at each field. From this data, the intensity of magnetization at saturation (Js) is determined by means of the following law[10] which defines the relationship between J, the intensity of magnetization and H, the field strength.

$$J = Js - \frac{a}{H} \text{ where a is a constant}$$

Once Js is determined experimentally, the ferrite content can be found out by the following relation[10].

$$\% \text{ Ferrite} = \frac{Js}{Js'} \times 100 \text{ where}$$

Js' is the intensity of magnetization at saturation for a 100% ferromagnetic sample containing delta ferrite. This Js' is not at all equal to the saturation magnetization of pure iron, but essentially depends on the chemical composition of the ferrite present in the weld metal[11]. Js' can be determined from the following formula[12].

$$Js' = 4\pi(1710 - 22.6 \times \% \text{ Cr} - 3 \times \% \text{ Ni}) \text{ Gauss}$$

Chemical analysis of the weld metal for Cr and Ni contents is necessary for this purpose.

This is a destructive method in that an all-weld specimen having specified dimensions of 4.6 mm. dia x 110 mm long is required to be magnetized. This method has advantages over other magnetic methods in that it is independent of the form and size of ferrite particles and when the volume of the sample is sufficiently large, it is also independent of ferrite distribution.

3. EXPERIMENTAL APPARATUS

Figures 1 and 2 show the schematic arrangement of the apparatus. While designing the apparatus, the recommendations

of ASTM Standard A342 - 64 "Methods to determine the permeability of feebly magnetic materials" have been followed wherever applicable. The apparatus consists mainly of the following.

(i) D.C. magnetizing solenoid: This is made of 5000 turns of 15 SWG copper wirewound on a former. This has a length of 500 mm. and a length/dia ratio of 6.

(ii) Test and compensating coils: These arewound on a glass tube of 8.3 mm dia and have 1527 and 2066 turns respectively. These are kept inside the magnetizing solenoid, with the specimen positioned inside the test coil.

(iii) Ballistic Galvanometer: This is used to measure the magnetic flux.

(iv) Three phase bridge rectifier: This provides d.c. power supply to the magnetizing solenoid.

(v) Standard solenoid: This consists of 500 turns of 17 SWG copper wire with a length/diameter ratio greater than 20. A search coil with 2000 turns is closely wound around the centre of solenoid. Power is supplied to this unit from a battery bank.

4. PROCEDURE

The value of effective area turns of the compensating coil is designed to be slightly higher than that of the test coil and this value is adjusted by means of variable resistance connected across it to obtain air flux compensation. This resistance is adjusted till a galvanometer connected to the two coils in series opposition does not defect upon reversal of the solenoid

current . After the above adjustment, the specimen is introduced inside the test coil and the deflection now obtained upon reversal of solenoid current is proportional to the intensity of magnetisation (J). The value of magnetic field (H) may be measured or assumed proportional to the solenoid current (I) because demagnetization due to end effects will be negligible for a feebly magnetic material[13].

The ballistic galvanometer used is calibrated by means of the standard solenoid circuit to obtain the proportionality between its deflection and the flux linking a search coil having known number of turns (i.e. Wb Turns).

The galvanometer deflection (d) is measured at six values of magnetic field (H) between 1700 and 2500 Oe after air-flux compensation. A graph is plotted between d and 1/H, i.e. between d and 1/ I by least square fit and extrapolated to 1/I=0. The corresponding galvanometer deflection viz. ds is proportional to the intensity of magnetisation at saturation, Js. Js is calculated from ds by referring to the galvanometer calibration and specimen diameter. From Js, per cent ferrite can be calculated as explained earlier.

5. RESULTS AND DISCUSSION

Measurements have been carried out on a large number of specimens using this apparatus. These have been compared with the measurements made by other available methods such as metallography and Ferritescope. Table-1 gives results of measurements for some of these specimens. The purpose of this comparison is not to evaluate the results of magnetic saturation method in terms of accuracy but rather to demonstrate the difference between various other methods and saturation method.

This method is independent of distribution of ferrite which may vary a great deal from point to point within a specimen. The variation in ferrite at different locations can be clearly observed in the results obtained by metallography and ferritescope. It will be noted that in most cases, ferrite values measured by magnetic saturation method are much less compared to those determined otherwise. The results of saturation method are more nearer to those predicted by DeLong diagram which also gives overall per cent ferrite of the specimen. The readings obtained with this apparatus agree quite closely with those reported by a French laboratory, using a similar set up. It may be remarked here that measurements on 4 specimens of type 347 weld metal deposited by electrodes from the same heat have given consistant results by the saturation method. In all these specimens ferrite was found to vary between 4.5 and 5.2% (See specimen No. 10 to 13 in Table-1).

The saturation method of determination of delta ferrite is an absolute method and does not need calibration [8], as it is based on measuring a fundamental property of the material of the sample. However, the present method makes use of certain equations for computing ferrite together with measurements of intensity of magnetization in the specimen. The intensity of magnetization at saturation is not actually measured experimentally because of the practical difficulty to produce saturation using very high magnetic fields. Instead, measurements of intensity of magnetization are made for field strengths, varying between 1700 and 2500 Oe, and from these the value of saturation intensity is obtained by the law of approach to saturation viz., $J = Js - \frac{a}{H}$. According to Sulmont et al [10] this law has been verified by several measurements made on different samples of Cr-Ni steels for magnetic fields varying between 1300 and 3000 Oe. It is noted that beyond 1300 Oe, the intensity of magnetization increases

linearly with field strength and hence extrapolation to saturation is valid. An error analysis was performed to take into account the errors occuring in different measurements, those associated with the calibration of standard solenoid, in the construction of the equipment and in the use of equations. It has been estimated that the results can be accurate to $\pm$ 1% ferrite or better, for ferrite contents lower than 5%.

The apparatus is currently used for ferrite measurement necessary for qualifying welding procedures as well as weld test coupons of components of the Fast Breeder Test Reactor.

6. ACKNOWLEDGEMENTS

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Table 1

COMPARISON OF FERRITE MEASUREMENTS BY DIFFERENT METHODS

Sl. No.	Weld metal	Magnetic saturation method		Ferritescope	DeLong Diagram	Metallography	Remarks
		RRC	French Laboratory				
1.	Batox D electrode of D & H type 316	1.5%	2%	0.5% to 7% Avg. - 2.4%	-	-	Ferritescope measurements made on 4.6 mm. dia rod; curvature correction applied. Arithmetic average of readings taken at 5 equidistant points on the 110 mm long specimen.
2.	Each electrode, 5 mm dia. batch No.4498 type 316	2.5%	3%	3.7% to 6.2% Avg. - 5%	-	-	
3.	Esab electrode, 3.15 mm. dia, batch No.14509, type 316	3%	4%	6.5% to 7.8% Avg. - 7.3%	-	-	
4.	INDX - D2 Advani Oerlikon electrode type 309	6%	7%	13.2% to 16.5% Avg. - 15%	-	-	
5.	Type 316	6%	-	-	6.5%	5.5%	
6.	Type 316	3%	-	7.7%	4%	7 to 10%	Another agency reported 4%, method not known.

Table 1 (contd.)

Sl. No.	Weld metal	Magnetic saturation method		Ferritescope	DeLong Diagram	Metallography	Remarks
		RRC	French Laboratory				
7.	Type 308	3%	-	5% to 8.4%	4%	6% to 9%	2% reported by manufacturer
8.	Type 308	2.7%	-	5% to 7%	-	16%	5% to 6% with Ferritector
9.	Type 308	3.1%	-	6% to 8%	-	10%	8% to 9% with Ferritector
10.	Type 347	4.6%	-	12% to 15% on round rod	8%	-	13.7% by another ferritescope
11.	Type 347	5.2%	-	14.2% to 16.5% on round rod	9%	-	17.3% by another ferritescope
12.	Type 347	4.5%	-	15% to 18% on round rod	8%	-	16% by another ferritescope
13.	Type 347	4.6%	-	16.5% to 18% on round rod	less than 8%	-	16.5% by another ferritescope

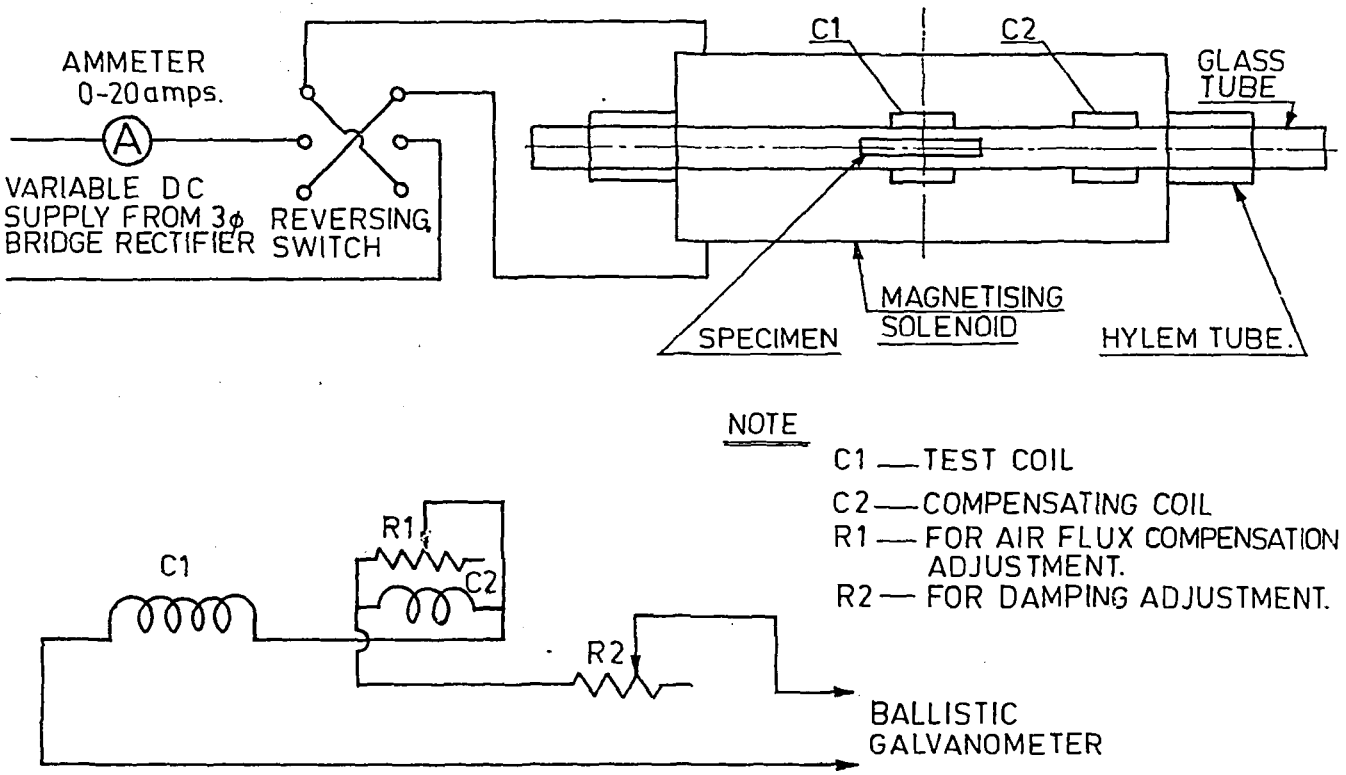
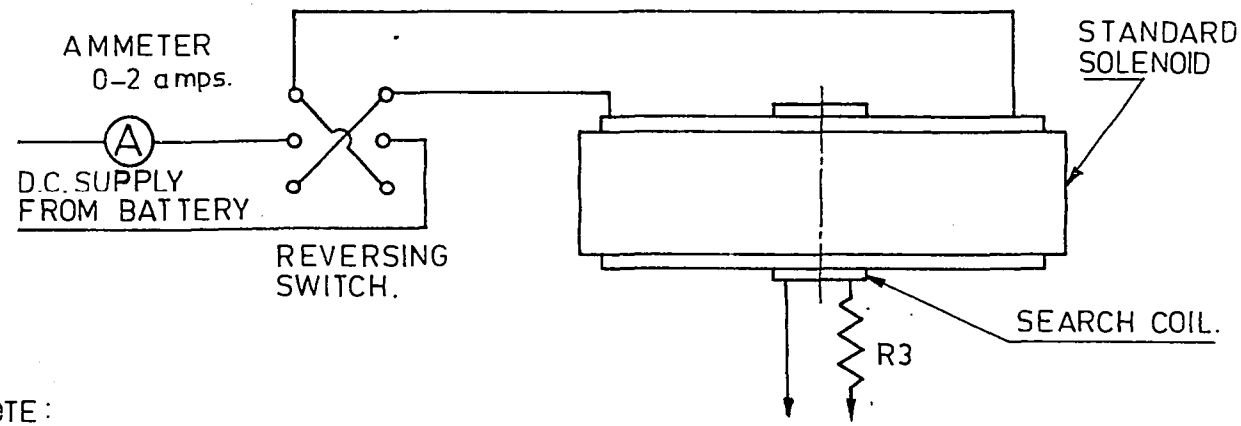


FIG.1 MEASUREMENT CIRCUIT.



NOTE:

R3 — FOR KEEPING CALIBRATION AND MEASUREMENT CIRCUIT RESISTANCES EQUAL.

FIG.2 CALIBRATION CIRCUIT.