Techniques and Standards for Measuring Ferrite in Austenitic Stainless Steel Welds

A new method of ferrite measurement is compared with the WRC recommended practice

BY A. W. BREWER AND R. L. MOMENT

ABSTRACT. A technique for constructing standards containing ferritic stainless steel in an austenite matrix is reported. Such a standard more closely resembles the metallurgical structure of a weld than either iron powder in a nonmagnetic matrix or various nonmagnetic coatings over a ferrite base.

Also, a different type of measuring instrument is evaluated which utilizes the interaction of magnetic particles with an alternating magnetic field. Data taken with this device are compared with those obtained using the more common magnetic type instruments. The new instrument has three advantages: (1) faster collection of data, (2) greater sensitivity to small point-to-point fluctuations, and (3) ability to be used on large and/or rough weld surfaces.

Introduction

The presence of delta ferrite in the range of 4-8% has proven effective in controlling microcracking of welds in austenitic stainless steels during cooling (Ref. 1). Partially ferritic cast stainless steels have improved welding characteristics and increased strength over purely austenitic alloys. On the other hand, more than 10% ferrite can contribute to a reduction in ductility and impact strength at low temperatures, a reduction of corrosion resistance in certain environments, and enhance formation of sigma phase at high temperatures (Ref. 1).

Thus it becomes extremely important to be able to measure the ferrite content in austenite at levels between 1 and 12%. Because no dependable technique for ferrite measurement has been found, the Welding Research Council (WRC) has adopted an arbitrary procedure which employs the Magne Gage and standards made with various thicknesses of a nonmagnetic coating over a magnetic substrate (Ref. 2). Ferrite levels are reported as ferrite numbers (FN) (Ref. 3). This standardization of measurement technique allows for better comparison of results from various laboratories. The FN is essentially equivalent to the ferrite content up to about 8%, but differs by increasing amounts above this level.

There are numerous methods for measuring the ferrite content of a weld, all of which, under certain conditions, may lead to incorrect results. They can roughly be classified into empirical, magnetic, and nonmagnetic categories. Empirical studies of the ferrite contents of welds as a function of nickel and chromium equivalent alloying constituents have established diagrams showing approximate ferrite content as a function of composition. The first such diagram was prepared by Schaeffler (Ref. 4), and has recently been improved by DeLong (Ref. 5) and Hull (Ref. 6) to include the effect of nitrogen and other elements.

Nonmagnetic techniques include x-ray diffraction, Mössbauer-effect and metallography. The latter one has been used extensively to check other methods, but is subject to error from (1) poor etching behavior of some alloys, (2) the counting technique employed, and (3) the aspherical shape of ferrite particles, particularly in welds. While variations on the order of 1% ferrite for levels up to 4%, and 2% for levels near 10% have been achieved, metallographic methods are time consuming and not applicable for a nondestructive test.

Measurement of the magnetic response of a weld has proved to be the simplest nondestructive method and is in widespread use. Two instruments are used in the majority of tests. The Severn Gage employs a balance beam and compares the attraction of a permanent magnet on one end to the weld and to different standards. This results in a range for the ferrite content bracketed by that of two standards. A more precise estimate is obtained by the Magne Gage. The force of attraction between a permanent magnet and the weld is measured against that applied to a lever arm by a spring. Values are obtained relative to the standards used to calibrate the spring force.

The weak link in these ferrite measuring techniques is calibration. The orientation of lenticular ferrite particles, which affects the magnetic response, will vary within a weld, and thus welds made using identical materials will not always give the same magnetic response. A homogeneous array of spherical ferrite in a homogeneous matrix will not necessarily give the same response as a weld having the same total ferrite content. However, this latter type of standard should be more easily reproduced than that made from weld beads.

As noted above, the WRC has

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adopted National Bureau of Standards standards comprised of varying thicknesses of a nonmagnetic coating over a magnetic substrate. These are to be used with the Magne Gage to calibrate it to the ferrite number scale. These standards bear no relation at all to the physical situation of ferrite in an austenite matrix, and cannot be used with an instrument such as the Ferrite Meter, described below whose readings are adversely affected by materials of high electrical conductivity such as copper or aluminum.

The present study was undertaken with two goals. The first was to evaluate an instrument which utilizes a different technique than the Magne Gage for detecting the presence of ferrite. The second was to prepare a standard of ferrite in austenite which would be more closely related to actual conditions existing in a weld, and which could be used with this instrument.

Ferrite Meter

Lassahn and Moment (Ref. 7) studied the suitability of using an eddy current instrument to detect ferrite in austenite. Pressed powder specimens of iron in Type 304 stainless steel and welds in this same alloy were used, and it was concluded that ferrite levels of 1% or more could easily be detected. A phase-sensitive eddy current system was used, with a probe having a nominal radius of 0.5 mm, and point-to-point ferrite variations within a weld were detected with good reproducibility. The spatial resolution of the system was 0.75 mm and the relative ferrite content was determined to within an estimated 0.04%. No good standards were available at that time to calibrate the system.

A commercial instrument made by the Institute Dr. Förster* operates on a similar principle. The Ferrite Meter, Model 1.053, is battery powered and uses a single-contact probe, the design of which is shown in Fig. 1. An alternating current is passed through the excitation coil which is wound into two sections. This induces a voltage in both parts of the receiver coil windings, which are connected in opposition. When the probe is kept away from a metal surface, the output voltage is zero. However, when the tip is brought into contact with an austenite-ferrite mixture, the magnetic field is disturbed owing to the presence of the ferrite. This is probably the same effect which influenced the eddy current probe in the earlier experiments.

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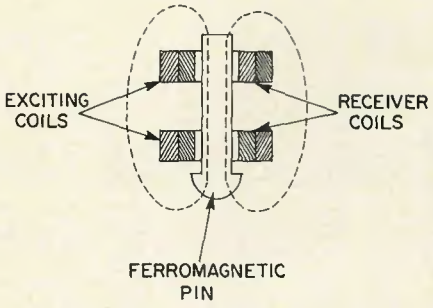

Fig. 1 — Ferrite Meter probe detail

The ferrite meter probe develops a voltage which depends on the amount of ferrite present, and this is read out on one of four scales covering 0 – 1, 3, 12 and 50% ferrite. Because the probe tip is hemispherical with a 0.75 mm radius, it makes a point contact with the surface to be measured, and thus can be used on rough welds and to study point-to-point variations across a weld. The volume of weld material influencing the readings has a radius of about 1.5 mm. This instrument would also be valuable for measuring ferrite in narrow welds such as those obtained using electron beam welding.

Standards

For any of the measuring methods, the accuracy of the ferrite value arrived at will depend on the calibration of the instrument and the volume of the weld actually measured. Weld pads have been created to be used as standards, though orientation of the ferrite stringers can change from point-to-point, and this makes it impossible to create two series identical to each other.

The standard included with the ferrite meter is supposed to represent 10% ferrite, and consists of iron powder mixed in a resin matrix. Point counting a photomicrograph of this coupon yielded 9.9%, while measurement with a Severn Gage gave greater than 14%, the highest range available with the Instrument.

It was concluded that a new set of standards which more closely approximated actual welds would be desirable. They should consist of ferrite in an austenite matrix with the compositions of each phase similar to those of actual welds. (It is important that the electrical conductivity of the standard be equal to that of a weld.) Powder metallurgy was chosen since it lends itself well to uniform mixing of two different phases.

The main problem with mixing powders has been obtaining truly uniform or homogeneous distribution of the components. In preparing mixtures of steel powders, the National Bureau of Standards found considerable trouble in obtaining uniformity using a "V" tumbler dry (Ref. 8). To avoid this problem it was decided to mix the powders wet in order to minimize agglomeration by static charging.

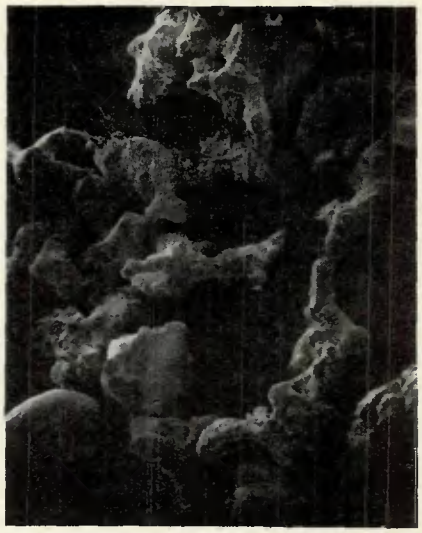

Fig. 2 — Scanning electron microscope photograph of type 316 stainless steel powder; X500, reduced 74%

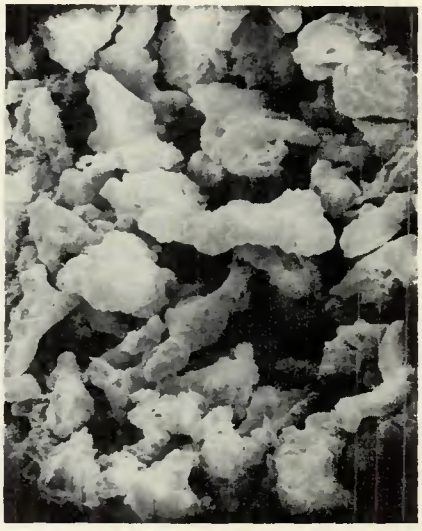

Fig. 3 — Scanning electron microscope photograph of type 430 stainless steel powder; X500, reduced 74%

Powders of +325 mesh 316L grade stainless steel were used for the austenitic, and 430 grade for the ferritic components of the mixtures. Two sets of five standards were prepared, with nominal compositions of 1.5, 3.5, 5.0, 7.5, and 10.0 percent ferrite. Each sample was mixed mechanically in a plastic vial using Freon TF as a vehicle. The final slurry had a consistency of wet mortar. After drying, standards were prepared from each mixture by pressing in a vacuum hot press for 30 minutes at 22,000 psi and

800 C. Longer pressing times or higher temperatures resulted in the precipitation of a third phase which contained manganese and silicon, as identified by electron microprobe analysis. Densities of the pressed samples ranged from 78.1 to 91.1 percent of the theoretical value.

Figures 2 and 3 show scanning electron microscope photomicrographs of the metal powders before pressing. They are essentially identical in shape and size. The uniformity of distribution of the 430 type stainless steel in the 316 type matrix can be seen in Fig. 4. A series of photomicrographs were taken of each standard to verify homogeneous dispersion of ferrite and also to use for point counting.

Ferrite Measurements

The ferrite content of each standard was determined by point counting, the Severn Gage, the Magne Gage, and the Ferrite Meter. Point counting was done on a minimum of two photomicrographs of each standard using a grid of 1032 points each time.

Results for both sets of standards were quite similar and are given in Table I. A plot of the data for one set is shown in Fig. 5. The Severn Gage brackets the ferrite content while the other two instruments read a value. All show a slightly nonlinear response (with the exception of the Severn Gage on the 7.5 percent standard), though it is apparent that their calibrations are not equivalent. The error in the Magne Gage readings is ±0.5 percent ferrite, as determined from data on the instrument and the graphs used to correct the dial readings to the ferrite values, while that for the ferrite meter is ±0.15 percent below 3 percent and ±0.6 percent up to 12 percent.

Eddy currents are sensitive to cracks or volds in a metal so there was concern that porosity in the pressed-powder samples might affect the readings. Accordingly, a series ot specimens was prepared from the same 5 percent mixture of type 430 in type 316 stainless steel. Varying pressures were used to obtain specimens ranging between 55 and 94 percent of theoretical density. Readings from the ferrite meter on these coupons are plotted versus density in Fig. 6, and while there is some slope to the curve, the variance between 75 and 95 percent density is less than 0.5 percent ferrite. Thus, it was concluded that the error associated with specimen density variations could be neglected.

Three additional powder metallurgy coupons were prepared using iron instead of type 430 ferritic stainless steel powder in order to study the effect of alloying on the magnetic response of ferrite. The data on these samples are listed in Table I and plotted in Fig. 7. The Ferrite Meter readings are about twice as high for the iron as tor the ferritic stainless, while the Magne Gage readings are slightly less than three times higher. It is important to note that the effect on the two instruments is not the same magnitude. However, one can see that the chemical composition of the ferrite in a standard will affect the calibration of ferrite measuring instruments and thus their ability to accurately measure ferrite in welds.

Table 1 — Data on Powder Pressed Ferrite Standards[a]

Composition	Series	Density, %	Point count, %	Severn Gage	Magne Gage	Ferrite Meter
1.5% 430 in 316	#2	81.7	1.49	0.5- 1.0	0.35	0.35
2.0% 430 in 316	#1	85.0	—	0.5- 1.0	0.3	0.5
3.5% 430 in 316	#1	83.0	—	1.5- 2.5	1.2	1.2
3.5% 430 in 316	#2	79.1	3.2	1.5- 2.5	1.1	1.0
5.0% 430 in 316	#1	79.0	5.0	2.5- 4.0	2.2	1.8
5.0% 430 in 316	#2	78.6	4.46	2.5- 4.0	2.1	1.5
5.0% Fe in 316		91.1	4.4	7.5-10.0	6.1	4.3
7.5% 430 in 316	#1	84.0	7.5	7.5-10.0	2.9	3.3
7.5% 430 in 316	#2	78.1	7.5	7.5-10.0	3.2	2.8
7.5% Fe in 316		90.5	7.3	12.0-15.0	9.6	6.5
10.0% 430 in 316	#1	82.0	—	7.5-10.0	5.0	4.4
10.0% 430 in 316	#2	78.6	10.2	7.5-10.0	5.1	4.2
10.0% Fe in 316		88.5	9.9	12.0-15.0	12.0	8.2

(a) Two series of standards were prepared using type 430 stainless steel powder mixed with type 316 stainless steel powder. Samples from each series are noted as #1 and #2. Three additional standards were prepared using powdered Fe instead of the type 430 stainless steel.

Fig. 4 — Micrograph of nominal 10% standard showing uniform ferrite (light particles) dispersion; X250, reduced 74%

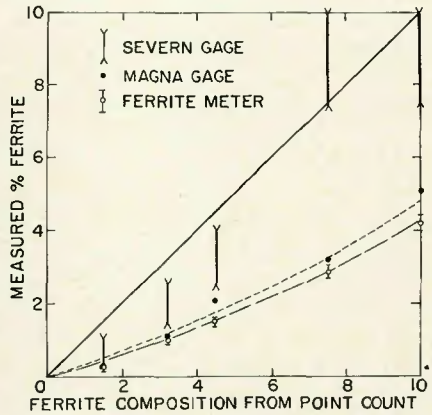

Fig. 5 — Measurements of ferrite in standards: values obtained using various instruments are plotted versus the value obtained from point counting

Discussion

Because the ferrite particles in the powder metallurgy standards are equiaxed, point counting is reasonably reliable for determining the actual concentrations. This leaves the Magne Gage and Ferrite Meter as the nondestructive measuring instruments to be evaluated. The Severn Gage is much less precise than these and thus is eliminated from further discussion.

The Magne Gage and Ferrite Meter have similar reading errors. On standards composed of type 430 ferritic stainless steel particles in a matrix of type 316 austenitic stainless, both instruments gave readings within 20 percent of each other. These values were about half of that obtained by metallographic point counting, and this discrepancy can be attributed to the calibration procedures specified for each instrument. Additional measurements were made on welds in type 304 and Nitronic 40 stainless steel (21-6-9).* The data, plotted in Fig. 8, show a good linear relationship with deviations only for data on

*Designation by Armco Steel Corporation.

welds. This is a result of the ferrite meter being able to read variations within a small volume of the weld and thus detect point-to-point changes while the Magne Gage averages over a larger area, the entire weld in this case. By relabeling the abscissa, direct conversion to ferrite numbers can be obtained.

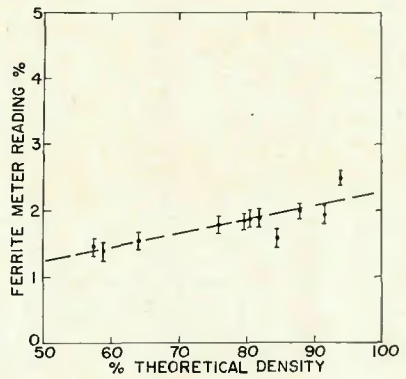

Fig. 6 — Ferrite meter reading versus pressed powder density for a nominal 5% ferrite standard

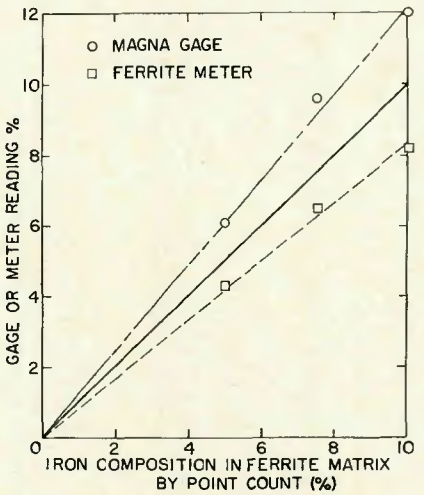

Fig. 7 — Magne Gage and Ferrite Meter readings versus point count on three iron powder-type 316 stainless steel standards

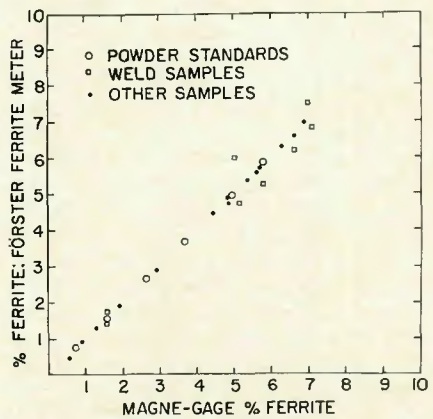

Fig. 8 — Ferrite Meter readings plotted versus those from the Magne Gage on a variety of samples. The data show that the Ferrite Meter measured regions of different ferrite content within the weld while the Magne Gage gave only a single average value

Alloying has a strong effect on the magnetic response of these instruments to ferrite. The extreme case considered in these experiments compared pure iron to an iron-chromium alloy, and differences by a factor of 2 to 3 were observed in the instrument readings.

Lesser compositional variations between ferrite in types 304, 308, and 347, for example, would be expected to have a smaller and perhaps negligible effect. But the calibration standards should consist of a ferritic alloy rather than pure iron.

Finally, the powder metallurgy standards prepared in this study lack the asymmetrical ferrite stringers found in weld structures. As such, they could not serve to calibrate an instrument used to make highly accurate measurements of ferrite in welds. However, they do give a closer approximation to the metallographic structure of a weld than either an iron powder standard or a coated ferritic plate. Variations from point to point in a weld make it difficult to know the ferrite distribution and level. However, while the Magne Gage averages the ferrite within a large area of the weld, the Ferrite Meter is able to detect smaller zones of higher or lower ferrite content. This is important when the distribution of ferrite in a weld is of interest, as in evaluating resistance to stress corrosion cracking.

Conclusions

1. Standards of ferrite in austenite can be made using powder metallurgy techniques, though they are more difficult to prepare than the coated standards currently specified by the WRC.

2. Ferritic stainless steel alloys give different responses than iron particles when measured using magnetic field instruments.

3. An eddy-current type of measuring instrument can easily detect ferrite in the range of 0-10 percent. It requires a calibration standard having the same electrical conductivity as weld metal and cannot be calibrated using the WRC coated standards.

4. The small probe size of the Ferrite Meter allows variations from point to point within a weld to be measured. It is possible to take readings on large and/or rough welds, or on small electron beam welds.

5. The Magne Gage and Ferrite Meter give readings within 20 percent of each other over the range of 0-10 percent ferritic stainless in austenite. The difference is larger when iron in austenite is being detected.

Acknowledgment

R. B. Fischer offered valuable advice on pressing the powders, and Dr. M. R. Harvey suggested the technique for mixing them. Thanks are also tendered to W. G. Thorvaldson for some comparative measurements made on weld specimens.

Discussion by H. C. Campbell

Moment and Brewer propose to make standards for ferrite measurement by powder metallurgy techniques from plus 325 mesh grains of 430 and 316L stainless steel, with a few samples of "iron" (no other identification) mixed with 316L. They admit at the outset that "A homogeneous array of spherical ferrite in a homogeneous matrix will not necessarily give the same response as a weld having the same total ferrite content," but then they forget all that in their enthusiasm about this "standard" being "more easily reproduced" than weld beads. It is equally true that NBS thickness standards "bear no relation at all to the physical situation of ferrite in an austenite matrix," but the authors overlook the fact that arbitrary Ferrite Numbers are internationally acceptable because they are traceable to pedigreed reference standards.

Nowhere have the authors tied their "standard ferrite" (the percent of 430 powder in their 316L compacts) to the true ferrite content of a weld. Not that it could be — the WRC Advisory Committee and the National Bureau of Standards have concluded that present knowledge does not permit determining the absolute ferrite content of a weld (Ref. 9).

The deviations in magnetic readings which bothered Moment and Brewer have already been explained in two major papers referenced in the articles they have quoted. Simpkinson and Lavigne (Ref. 10) and Fleischmann (Ref. 11) concluded that there can be no consistency in magnetic readings unless made at magnetic saturation.

Since the Magne Gage and Severn Gage do use strong magnets, the broad area which they sample becomes an advantage in determining average Ferrite Numbers. Because of the strength of these probes, point to point variations in weld ferrite are averaged out, whereas the Forster Meter reports a confusing mass of readings which the operator must average.

To summarize, three comments seem pertinent.

- The authors' primary conclusion will become valid when their powder compacts are calibrated in Ferrite Numbers.
- The German instrument they de-

scribe will be an excellent tool when calibrated by the WRC procedure (AWS A4.2-74). This procedure fully describes the use of secondary standards necessary to calibrate such instruments.

- This discussion will serve a valuable purpose if it steers the USERDA and others away from powder metal compacts divorced from the FN concept.

Discussion by W. T. DeLong

The authors have made a worthwhile contribution to the very extensive literature in this complex and controversial field. It is necessary and desirable to relate their work to the conclusions reached by the two major committees in the United States and Europe on this topic over the past 15 years, even though the discussion must necessarily be brief.

The calibration of magnetic instruments for ferrite measurement should be done following AWS A4.2-74 (Ref. 12). It is an extension of the WRC system described briefly in this article, and provides for NBS coating thickness standards for calibration of Magne Gages and weld metal standards for calibration of other instruments.

The International Institute of Welding has accepted this WRC concept and committed itself to establishing a procedure which will result in values for Ferrite Number on weld metals equal to those produced by instruments calibrated with the WRC/AWS procedure.

The first problem faced by the WRC and IIW committees was that there were literally dozens of different calibrations of "percent ferrite." Round robins showed that the spread in these was roughly plus 60% minus 40% from the mean, a very substantial spread! In order to avoid a situation in which the WRC was pushing just another "percent ferrite," one which could easily be confused with dozens of other different scales which were also percent ferrite, the WRC committee decided to replace percent ferrite with the term Ferrite Number, this number representing the true percent ferrite in weld metal to the best of our ability to establish it at the time.

The Ferrite Number scale established by the WRC was set so as to be approximately equal to the mean percent ferrite reported for the specimens studied in extensive United States and European round robin tests on a series of weld metals. Subsequent work established that the values selected were in fact reasonably representative of the best true percent ferrite, at least up to about 10% ferrite, as established by two careful and independent pieces of work (Refs. 9, 13). The first reference was based upon magnetic saturation tests on European weld metals used in one of their round robins, and the second upon Mossbauer-effect examinations by the United States National Bureau ot Standards of some weld metal specimens used in the United States round robin. Thus while the present calibration standards may be labeled as arbitrary, they are specifically based upon the best available data on the true ferrite content of weld metals.

Another key question on instrument calibration considered by the WRC committee was where and how to obtain dependable and guaranteed primary standards for calibration over a period of years. The Committee decision was that the NBS coating thickness standards meet these criteria. Standards effectively derived from this basic starting point, such as weld metal standards rated for Ferrite Number, or powder metal compacts if they should become available and are carefully rated in Ferrite Number and are proved suitable based on adequate testing, would also be satisfactory.

The authors' proposed powder compacts must also be considered arbitrary standards since the morphology of the magnetic particles in the compacts is quite different from that of weld metals. The authors' Table I illustrates the very substantial difference in magnetic response of a calibrated Magne Gage from the volume percent of magnetic powder in the specimen; i.e., 5% by weight of 430 powder in 316 is read as 2.2 and 2.1 Ferrite Number by the Magne Gage. This confirms work by Simpkinson back in 1949 through 1952 (Refs. 10, 14) and by DeLong et al., in 1956 (Ref. 15).

One question which must be asked is whether powder metal compacts will be made, tested, sold, and guaranteed to a specific Ferrite Number based upon the WRC/AWS procedure, over a period of time by a dependable source.

A second and related question is whether such standards, which must be regarded as secondary standards since their assigned FN values would be derived from tests on Magne Gages calibrated to the AWS procedure, are as good as secondary weld metal standards similarly rated for FN. As the authors point out, compacts are not like weld metal, and instruments do differ in response. Thus, while weld metal must respond as weld metal on any and all instruments, a compact will not necessarily show the same shift in response as a weld metal when tested on a Magne Gage and then Instrument X. The result could be an incorrect calibration of Instrument X with powder compacts. It would be necessary to prove that this is not a problem before accepting or recommending metal powder compacts as standards to calibrate other instruments.

With regard to the conclusions reached by the authors, the information available from committee work and personal experiences leads me to agree with conclusions 1 and 2. With regard to conclusions 3 through 5, some cautions are in order. The instrument proposed can undoubtedly be very satisfactory if properly calibrated and periodically rechecked against weld metal standards rated in terms of Ferrite Number. If not, it may or may not read correctly. Three or four Magnetoscopes produced by the Institute Dr. Förster, which were tested in Europe in a round robin years ago, gave widely different "percent ferrite" readings on a given set of weld metals. The basic use of the instrument at that time was for measuring permeability. I believe that this is the same instrument, since adopted to measure ferrite and named the Ferrite Meter. It is described and discussed in a 1968 IIW Document (Ref. 16). It seems generally suitable for measuring the ferrite content of weld metal, *provided* that it is properly calibrated to do so. The IIW document reported that the volume of weld metal sensed by the probe had a radius of about 10 mm. This would conflict with the author's conclusion 4. However, it is very possible or even probable from a review of the authors' article versus the IIW Document (Ref. 16) that the probe has been reduced in size and that it now responds to a smaller volume of weld metal.

H. C. CAMPBELL is a member, and W. T. DeLONG is Chairman of the Advisory Sub-Committee of the High Alloys Committee of the Welding Research Council.

Author's Reply

I wish to thank the reviewers, Dr. H. C. Campbell and W. T. DeLong for their comments. I agree that the subject is complex and there is a need to be extremely careful in developing standards and in calibrating instruments. The intent of this paper was to present some additional information which could be of benefit to people working in the field. As printed, the paper contains some changes made to conform to some of the criticism.

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