



Contents lists available at ScienceDirect

## Journal of Nuclear Materials

journal homepage: [www.elsevier.com/locate/jnucmat](http://www.elsevier.com/locate/jnucmat)

## Study on the weld characteristics of 316LN by magnetization measurement

Hyoung Chan Kim<sup>a,\*</sup>, K. Kim<sup>a</sup>, Y.S. Lee<sup>a</sup>, S.Y. Cho<sup>a</sup>, H. Nakajima<sup>b</sup><sup>a</sup> National Fusion Research Institute, 52 Yeoeun-Dong, Yusung-Gu, Daejeon 305-333, Republic of Korea<sup>b</sup> ITER Superconducting Magnet Technology Group, Japan Atomic Energy Agency, Mukoyama, Naka-shi, Ibaraki-ken 311-0913, Japan

## A B S T R A C T

We studied the characteristics of 316LN welded joints produced by different welding fillers through temperature and field dependent magnetization measurements. The magnetic permeability of welded joints showed significant variations depending on the used welding fillers. Qualitative changes from 316LN base material were observed in the field dependent magnetization of welded specimens. While the self-welded and low-Mn content filler-welded samples showed signs of secondary phase formation, considerable suppression of the secondary phase formation was observed in high-Mn content filler-welded samples. The austenitic-structure-retaining property was also observed in the FE-SEM microstructure.

© 2008 Elsevier B.V. All rights reserved.

## 1. Introduction

Since materials in fusion reactors are subject to physical constraints such as electromagnetic, thermal and mechanical loads depending on their operating conditions, development and characterization of relevant materials satisfying the requirements is a key milestone for the expected performance of fusion reactors. For the structural material of superconductor magnet system in fusion reactors, high mechanical strength at cryogenic temperature is the primary concern and austenitic stainless steels such as 316LN, JJ1, EC1 and JK2LB are used for the cryogenic applications [1–3]. 316LN, one of the austenitic steels, is considered to be used for ITER TF magnet structure and conductor conduit material [4,5]. While the crystal structure of an austenitic stainless steel is stabilized in FCC at the temperature 900–1400 °C, it tends to undergo a structural transformation into BCC metals such as ferritic or martensitic steel during cooling below 900 °C. Because BCC metals show brittle behaviour at low temperature due to the ductile-to-brittle transition [6,7], the welding technique of 316LN steel to prevent the BCC metal formation and the relevant characterization of the welded joint are required [8]. In ITER, the use of high-Mn content (6–10%) welding filler such as DIN1.4455 and JJ1 is required for TIG welding of 316LN. In order to confirm the performance of suggested welding fillers, proper comparative evaluations of the welds out of the welding fillers should be made.

In this work, we studied the characteristics of 316LN welded joints produced in different welding conditions. Since the ferritic or martensitic phase that could be formed in welding has higher magnetic susceptibility, quantitative comparison on the formation

of secondary phases such as ferrite can be made by magnetization measurement. Among the physical properties that could be employed as an indirect method for the non-destructive tests, the magnetic property is one of the most sensitive properties to estimate the impurity inclusion.

For 316LN base material, 316LN plates (by DAIDO Steel) were used and four different welding conditions were produced by using three kinds of welding fillers. We present our results of magnetization measurement of 316LN base material and welded joints in the temperature range from 300 K down to 4 K and in magnetic field up to 2 T. For a complementary analysis of the welded joints, FE-SEM micrographs were taken to compare the microstructures of the specimens. The effect of using high-Mn content welding wire on the weld property is discussed based on the results.

## 2. Experimental details

TIG (Tungsten Inert Gas) welding was employed to produce the welded joint. Welding was done in flat position on the two 316LN plates of 2 mm thickness placed side by side under restraint. The chemical compositions of 316LN base material and three welding filler materials used in this study are shown in Table 1. The notable difference in the chemical composition is Mn content as was mentioned in the Introduction. The Mn content of DIN1.4455 and JJ1 fillers are 7.32% and 9.8% which are appreciably higher than those of ER316L filler and 316LN base material. The self-welded (autogenous) sample was produced by welding without using any welding filler. Three filler-welded samples were produced by welding using three kinds of welding filler, one is normal ER316L and the others are high-Mn content welding fillers, DIN1.4455 and JJ1. With the other welding conditions being the same, we can single out the effect of welding filler on the weld property through quantitative comparisons.

\* Corresponding author.

E-mail address: [chankim@nfri.re.kr](mailto:chankim@nfri.re.kr) (H.C. Kim).

**Table 1**

Chemical compositions (wt%) of 316LN base material and three filler materials (ER316L, DIN1.4455, JJ1) used in this study.

Material	C	Si	Mn	P	S	Ni	Cr	Mo	N
316LN	<0.02	<0.75	<2	<0.045	<0.03	10.0–14.0	16.0–18.0	2.0–3.0	0.16
ER316L filler	0.03	0.30–0.65	1.00–2.50	0.03	0.03	11.0–14.0	18.0–22.0	2.0–3.0	
DIN1.4455 filler	0.01	0.16	7.32	0.004	0.005	15.84	18.65	2.82	0.15
JJ1 filler	0.02	0.43	9.8	0.008	0.009	14.04	12.07	4.8	0.14

For the magnetization measurement the 316LN material was cut into rectangular samples of the optimal size for the precise magnetization measurement. For the welded 316LN, electric discharge wire cutting method was used to cut into rectangular samples around the welded joint region. After cutting, mass and dimensions of the samples were measured. The dimensions, mass and density of the specimens used for the measurements are shown in Table 2. Fig. 1 shows top view of the 316LN welded joint part and the specimen cutting configuration. The rectangular cut in the joint centre is for the magnetization measurement.

Magnetization was measured using MPMS (Magnetic Property Measurement System, Quantum Design™). A sample is 4 cm transported through the 2nd derivative type pick-up coil and the induced signal by the magnetic moment is transferred to the SQUID sensor. Temperature dependent magnetic susceptibility was measured from 300 K down to 4 K and field dependent magnetization was measured from  $-2$  T to 2 T. The microstructure of the samples was obtained by FE-SEM. To get clear micrographs, surface etching of the samples was carried out with etching solution (DI water 150 ml + KOH 30 g +  $K_3Fe(CN)_6$  30 g) heated to 70 °C.

**Table 2**Magnetic permeability  $\mu$ , specimen dimension, mass, and density of 316LN base material and welded joints ( $\mu$  is determined at  $H = 4000$  Oe).

Sample	$\mu@300$ K	$\mu@4$ K	Dimension (mm)	Mass (mg)	Density (g/cm <sup>3</sup> )
Base metal	1.00258	1.00693	$2.2 \times 2.45 \times 4.05$	155.61	7.13
Self-weld	1.0451	1.0538	$1.95 \times 2.6 \times 3.95$	157.05	7.85
ER316L filler-weld	1.0406	1.0498	$1.95 \times 2.95 \times 4.2$	186.31	7.71
DIN1.4455 filler-weld	1.0173	1.0234	$1.95 \times 2.85 \times 3.95$	171.17	7.8
JJ1 filler-weld	1.0187	1.0247	$2.0 \times 2.75 \times 4.0$	169.0	7.68



**Fig. 1.** Top view of 316LN welded part and the specimen cutting configuration. The picture shows the welded joint produced by DIN1.4455 filler and the dimension of the rectangular cut is 3.95 mm  $\times$  2.85 mm.

### 3. Results and analysis

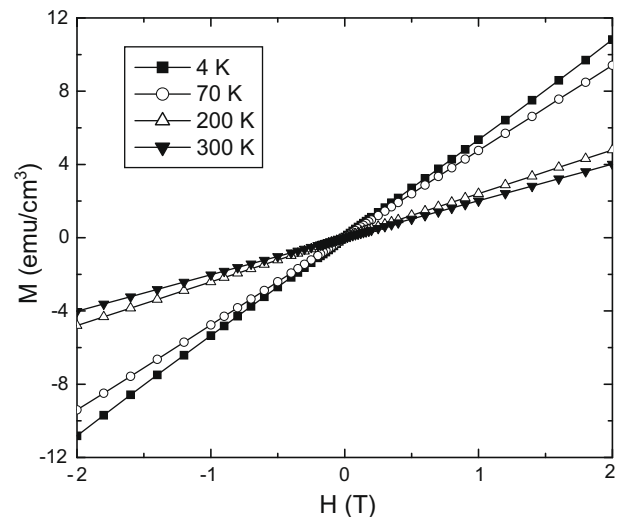
#### 3.1. Comparison of magnetic permeability at 300 K

Since we obtain the magnetic moment  $m$  [emu] of a sample by measurement using a magnetometer, we get the sample's magnetization  $M$  [emu/cm<sup>3</sup>] dividing the magnetic moment by sample's volume. The magnetic susceptibility  $\chi$  is the magnetization divided by the applied field intensity  $H$  and the magnetic permeability  $\mu$  is obtained through the relation  $\mu = 1 + 4\pi\chi$ . The magnetic permeabilities measured at 300 K and 4 K of the five samples are listed in Table 2.

The magnetic permeability of 316LN base material at 300 K is 1.00258 confirming its nearly non-magnetic behaviour. However, the magnetic permeabilities of the self-welded and ER316L filler-welded samples are 1.0451 and 1.0406, respectively, indicating 17 times increase of the magnetic susceptibility at the welded part compared to the parent material. On the other hand, the magnetic permeability 1.0173 of DIN1.4455 filler-welded sample turns out to be lower than the 1.05 engineering criterion [5]. Since the field dependent magnetization of welded samples show non-linear behaviour at low field (see Fig. 4), the nominal magnetic permeability determined at lower field tends to increase.

#### 3.2. Temperature and field dependence of magnetization

Fig. 2 shows the field dependence of magnetization of 316LN base material measured at different temperatures from 300 K to 4 K. Though a minute non-linearity is observed at low field below 2000 Oe, the magnetization becomes linear with field above 2000 Oe indicating paramagnetism of the material. Fig. 3 shows the temperature dependence of the magnetic susceptibility of 316LN base material measured at 4000 Oe. We can see Curie–Weiss type behaviour of susceptibility above 100 K. An interesting



**Fig. 2.** Magnetization of 316LN base metal measured at different temperatures from 4 K to 300 K.

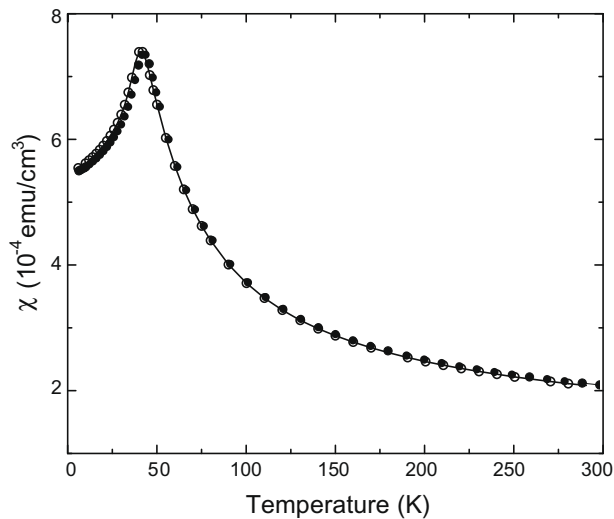


Fig. 3. Temperature dependence of 316LN base metal measured at 4000 Oe. The magnetic permeability at 300 K is  $\mu = 1 + 4\pi\chi = 1.00258$ .

feature in the temperature dependence is the susceptibility has a maximum around 40 K and decreases at lower temperature.

Fig. 4(a) shows the field dependence of magnetization of four welded joint samples of 316LN measured at 4 K. Compared to the paramagnetic linear behaviour of 316LN parent material, the magnetization of the self-welded and ER316L filler-welded samples show ferromagnetic-like rapid increase at low field below 2000 Oe and the magnetization becomes linear at high field. From this result, we can infer the formation of secondary phases such as the ferromagnetic ferritic or martensitic phase out of the austenitic structure at the welded region. While the magnetization of the welded sample produced with high-Mn content welding wire, DIN1.4455 and JJ1, shows ferromagnetic-like behaviour similar with the previous two samples, the reduced emergence of the behaviour indicates suppression of the secondary phase formation out of the austenitic structure. In the field dependence of magnetization of the four samples measured at 300 K (not shown here), we can see similar behaviour of the strong suppression of ferritic ferromagnetic phase formation in the DIN1.4455 and JJ1 filler-welded samples. Field dependent magnetization of the DIN1.4455 filler-welded sample measured at four selected temperatures is shown in Fig. 4(b). The result shows that the magnetic property of the weld is not changed qualitatively over the temperature range from 300 K to 4 K though the magnetization increase at high field is a little diminished at higher temperature.

Temperature dependent magnetic susceptibility of the four weld samples is shown in Fig. 5. Above 100 K, they show Curie–Weiss type increase in susceptibility with lowering temperature and reach a maximum around 40 K and then decreases at lower temperature. The estimated susceptibility ratio  $\chi(4\text{ K})/\chi(300\text{ K})$  from the result is from 1.2 to 1.33, which indicates that the magnetic property of the welds does not show a drastic change over the measured temperature range. Though all the four samples show similar behaviour with temperature, the magnitude of the susceptibility is decreased in accordance with welding method from self-welding and normal filler-welding to high-Mn content filler-welding. Since the DIN1.4455 filler-welded sample and JJ1 filler-welded sample do not show a notable difference in the magnetic property, additional evaluations are required to compare the welding performance of the two filler materials. Recent side bend test on the narrow gap TIG welding of 100 mm thick 316LN and JJ1 plates showed some micro-cracks in DIN1.4455 filler-welded sample [9].

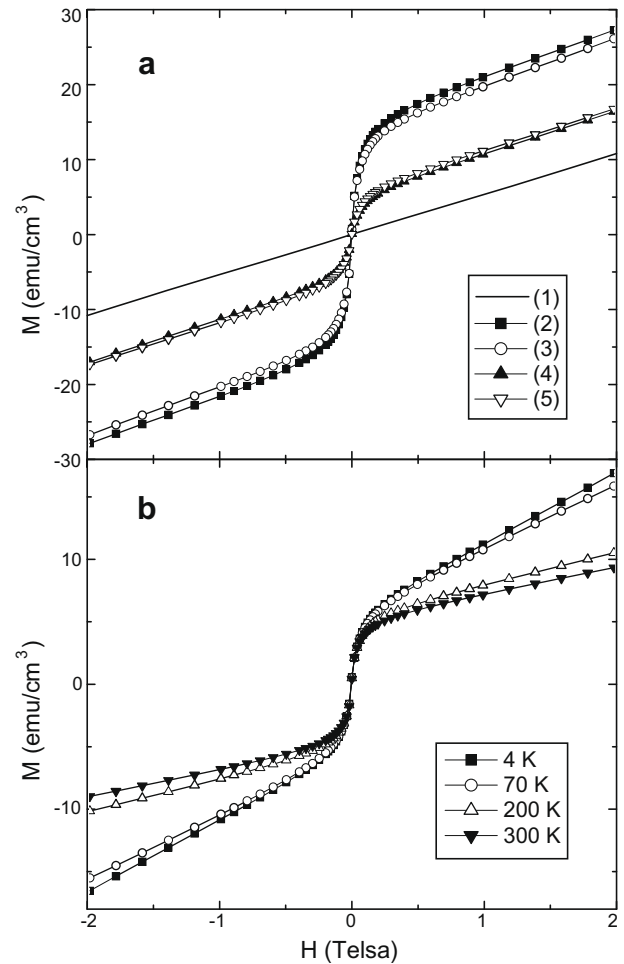


Fig. 4. (a) Magnetization of 316LN base metal and weld specimens measured at 4 K. (1) Base metal, (2) self-weld, (3) ER316L filler-weld, (4) DIN1.4455 filler-weld, and (5) JJ1 filler-weld. (b) Magnetization of DIN1.4455 filler-weld sample measured at different temperatures from 4 K to 300 K.

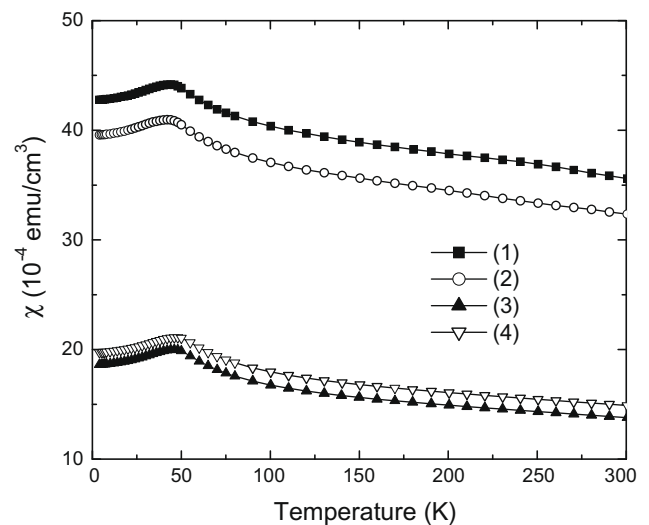
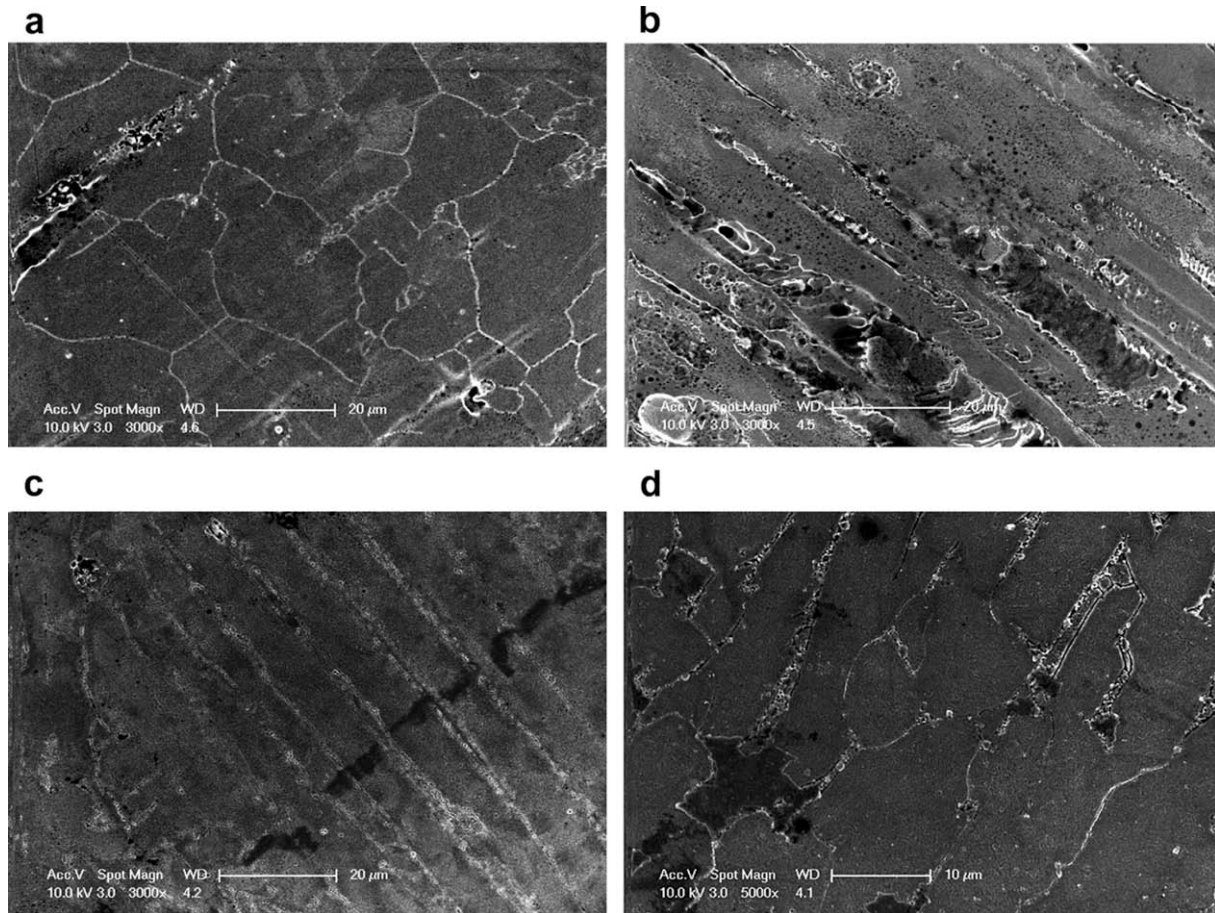


Fig. 5. Temperature dependence of magnetic susceptibility for four welded 316LN specimens, (1) self-welded, (2) welded with ER316L, (3) welded with DIN1.4455, and (4) welded with JJ1 filler.



**Fig. 6.** SEM micrographs showing the microstructure of 316LN (a) base metal, (b) self-weld, (c) ER316L filler-weld, and (d) DIN1.4455 filler-weld samples.

### 3.3. Comparison of the microstructure

Microstructures of 316LN base metal and weld samples are shown in Fig. 6. In 316LN base metal, we can see well shaped domains separated by clearly visible grain boundaries which characterize the austenitic structure [10,11]. The grain size ranges from about 10  $\mu\text{m}$  to 30  $\mu\text{m}$ . In self-welded 316LN sample, quite elongated domain structures are observed that differs from those of 316LN base metal. We can see sign of precipitation and significant melted micro-slag along the grain boundaries. While the microstructure of ER316L filler-welded sample is similar with that of self-welded sample, it shows reduced micro-slag along the grain boundary. Contrary to the previous two weld samples, DIN1.4455 filler-welded sample shows the austenitic microstructure like 316LN base metal. Although minute sign of precipitation is observed along the grain boundary, we can see the characteristic domain structures that were observed in 316LN base metal. The microstructure observation supports the previous results of magnetization measurement that high-Mn content filler-welded sample retains more austenitic properties of 316LN than the other two weld samples.

### 4. Conclusion

We investigated the weld characteristics of 316LN produced by TIG welding using different welding fillers. Through magnetization measurements, we estimated and compared the degree of secondary phase formation during welding. We have found the use of high-Mn content welding filler such as DIN1.4455 and JJ1 is effective to prevent the formation of the brittle ferritic phase at the

welded region. The microstructure comparison supported the result of magnetization measurement that high-Mn content filler-welded 316LN specimens retain austenitic property. The result of our experimental study clearly showed the usefulness of magnetic characterization for a quantitative evaluation on the impurity phase inclusion in welded joints. In order to compare the welding performance of DIN1.4455 and JJ1 fillers, further experimental studies in addition to the magnetic characterization on the welded joint are required.

### Acknowledgement

This work was supported by Fusion Technology Research Fund of National Fusion Research Institute and by the Ministry of Science and Technology of Korea under the ITER Project Contract.

### References

- [1] L.T. Summers, J. Fus. Energy 11 (1992) 39.
- [2] L.T. Summers, J.R. Miller, J.R. Heim, Adv. Cryo. Eng. Mat. 36 (1990) 769.
- [3] K. Hamada, H. Nakajima, K. Kawano, K. Takano, F. Tsutsumi, K. Okuno, T. Suzuki, N. Fujitsuna, Cryogenics 47 (2007) 174.
- [4] 'ITER Final Design Report', ITER IT Design Description Document, January, 2004.
- [5] 'Specification and Performance Database for Steel Jackets for Nb<sub>3</sub>Sn Conductors', ITER Design Description Document, July, 2005.
- [6] J.W. Morris Jr., Adv. Cryo. Eng. 32 (1985) 1.
- [7] S.J. Zinkle, APS Division of Plasma Physics 6th Annual Meeting, 2004.
- [8] T.A. Siewert, C.N. McCowan, Adv. Cryo. Eng. Mat. 38 (1992) 109; D.J. Alexander, G.M. Goodwin, Adv. Cryo. Eng. Mat. 38 (1992) 101.
- [9] K. Niimi, H. Nakajima, in: Korea–Japan Collaboration Meeting on the ITER Superconducting Magnet, Ulsan, September 2007.
- [10] ASTM E112, Standard Test Methods for Determining Average Grain Size.
- [11] M. Sireesha, V. Shankar, S.K. Albert, S. Sundaresan, Mater. Sci. Eng. A292 (2000) 74.