Abridged version

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The body can be viewed from the next page.

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1 Introduction

Particle physics has been making remarkable progress in recent years, with theory and experiment developing interdependently. In particular, experimental research using the synchrotron-type particle accelerator as a powerful tool is most essential in high-energy physics. The accelerator itself, designed for higher performance, is made larger in size. To meet all these requirements, the application of superconductivity technology has already become a reality. Magnets for use in orbit-bending and focussing of protons and electrons are now being produced with superconductivity technology, with the use of superconducting cavity started already.

As superconducting magnets for accelerators, "Tevatron" magnets are already in use at Fermi National Accelerator Laboratory (FNAL) in the U.S. (**Photo 1**),[1] and have also been adopted in the "HERA" project of Deutz Synchrotron Laboratory and in the "LHC" project of CERN, where its construction has started. Furthermore, a bigger project, the so-called Superconducting Super Collider (SSC) Project is now in progress in the U.S.

In the development and manufacture of large superconducting magnets with higher magnetic field, the following are required, viewed from the material side:

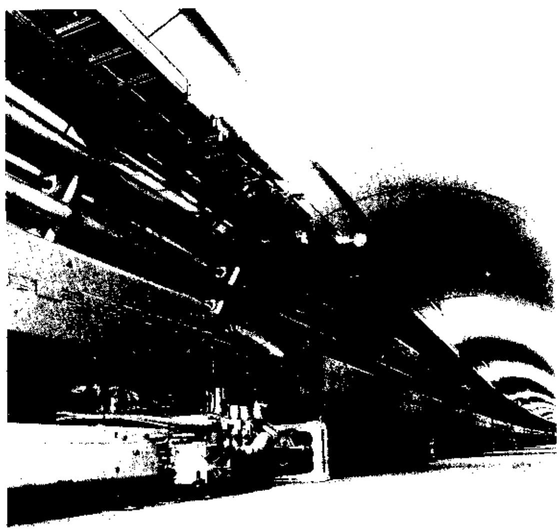

Photo 1 Superconducting magnets installed in tunnel for accelerator (Photo from a pamphlet of Fermi National Accelerator Laboratory)[1]

* Originally published in *Kawasaki Steel Giho*, 21(1989)3, pp. 245-249

(1) High-performance, high-reliability superconducting cable
(2) Cryogenic, high-strength non-magnetic supporting material
(3) High-magnetic yoke material for cryogenic magnetic shielding
(4) High-function heat insulating material

This paper describes the results of research and development of the cryogenic supporting material (2) mentioned above. The research targets are outlined below.

(1) To minimize magnetic fluctuation in the bore tube, permeability μ at 4 K is to be set at 1.002 or below.
(2) To withstand the localization of electromagnetic force and pre-stress, yield strength at the cryogenic temperature (4 K) and room temperature (RT) are to be increased ($\sigma_{y(4\,K)} \geqq 1.200$ MPa and $\sigma_{y(RT)} \geqq 620$ MPa).
(3) Anticipating the use of intermetallic-compound-type Nb_3Sn superconducting cables, deterioration of cryogenic ductile toughness after precipitation heat treatment at about 700°C is to be suppressed.
(4) Satisfactory fabricating characteristics are to be specified for practical use.

2 Test Method

To attain the above-mentioned targets, it was decided to examine Fe-Mn austenitic steel, because austenitic stainless steel poses a problem with cryogenic magnetism and it was considered difficult to make non-magnetism compatible with high strength. Besides, aluminum, copper and titanium have their own merits and demerits.

In preparing samples, 50-kg and 5-t ingots were made in a vacuum induction furnace, and after rolling them into slabs 20 mm or 100 mm thick, 5-mm-thick hot-rolled plates were manufactured. In the present experiment, the composition of such elements as Mn, C, N and V was changed. These plates were cold rolled on the Sendzimir mill into 1.5-mm-thick sheets (in the case of the 5-ton ingot). At the final process, temper rolling was carried out to render higher strength.

Using samples with a thickness of 20 mm, 5 mm and 1.5 mm obtained in this way, various tests were conducted at 4 K and room temperature. Namely, magnetic properties were tested in a magnetic field of 5 kOe given within a temperature range of 4 to 400 K, using a vibrating sample magnetometer (VSM). The uniaxial tensile test was performed by the Instron-type automatic tensile testing machine, and the Charpy impact test by using a specimen with sectional shape of 10 mm × 10 mm, at cryogenic temperature as well as room temperature.

In Charpy impact test, the specimens were cooled down by directly introducing liquefied He from the container onto the specimens wrapped up with heat-insulating material. Time required for cooling the V-notch portion of the specimen from room temperature to 4 K was about 3 min. For the stamping and spot-welding tests, a sheet testing machine was used, and tests were conducted at room temperature.

3 Test Results and Discussions

3.1 Composition and Magnetism

As the synchrotron accelerator becomes larger in size, namely, as its accelerating energy increases, it must bend and focus higher-speed particles; hence it is necessary to increase the magnetic field generated in the magnet. In the case of the above-mentioned "SSC" in the U.S., accelerating energy of oppositely-travelling protons is considered to be 20 000 GeV (or 20 TeV) for each, with a 6.6-tesla requirement for the magnetic field to be generated[2]. Since it is necessary to suppress the fluctuation of the large magnetic field to the order of 10^{-4}, it is required that the specific permeability of the non-magnetic collar supporting material be 1.002 or below (for intra-magnet magnetic field uniformity) at 4 K, and the dispersion of permeability among materials be 0.0005 or below (for inter-magnet magnetic field uniformity).[3]

To satisfy such requirements, the relations between the basic composition of Fe-Mn-based austenitic steel and specific permeability at 4 K were examined as mentioned earlier. The result is shown in **Fig. 1**. When specific permeability μ was measured at 4 K on specimens subjected to a 10% temper-cold-rolling in anticipation of strength increase after full annealing, the values of specific permeability were divided into three regions of $\mu \geqq 1.02$, $\mu \leqq 1.002$ and $1.02 \geqq \mu \geqq 1.002$ as shown in the figure, depending upon changes in the composition of Fe-Mn-C. Then, in addition to the above-mentioned criterion of $\mu \leqq 1.002$, the deterioration of hot workability due to an excess amount of Mn and that of machinability due to an excess amount of C were taken into account, and the region shown in Fig. 1 was specified as a desirable composition range of Fe-Mn-based steel. Based on this, a synthetic analysis was made of physical and mechanical properties and usability. The authors found a high-Mn steel shown in **Table 1** as a newly developed non-magnetic material for accelerator superconducting magnet supporting collars. Its composition features low C-high Mn as a matrix and additions of Cr, Ni, V and (Al and/or Ca).

The newly developed high-Mn steel was compared with 316LN, a typical cryogenic austenitic stainless steel, in terms of temperature dependence of specific permeability μ (both materials are in a full annealed state, with a 5 kOe magnetic field applied). **Figure 2** shows the results.

Since 316LN has a magnetic transformation point (Néel temperature T_N) near 25 K, μ shows significant

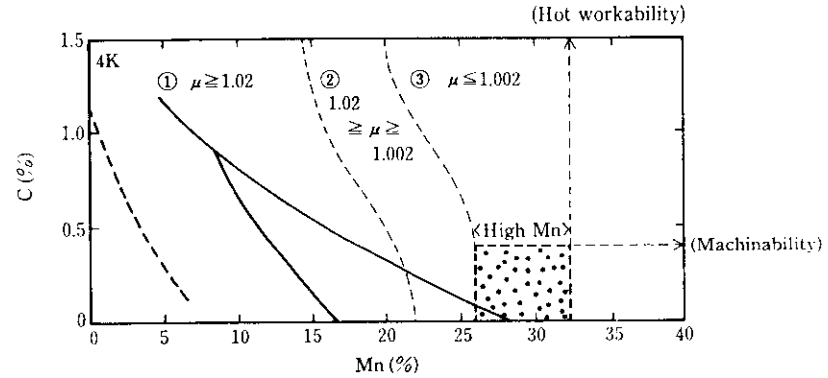

Fig. 1 Newly developed high Mn steel on the Fe-Mn-C phase diagram at 1 100°C (Permeability μ was measured at 4 K on specimens temper cold rolled by 10% after full annealing.)

Table 1 Chemical compositions of newly developed high Mn steel and 316LN stainless steel for reference (wt.%)

Steel		C	Si	Mn	P	S	Cr	Ni	N	V
High Mn*	Nominal	≦0.4	≦1.0	26~32	≦0.04	≦0.01	6~8	0.5~1.5	0.05~0.15	0.05~1.0
	Example	0.12	0.6	27.9	0.035	0.007	7.0	1.5	0.09	0.06
316LN (Reference)		0.03	0.5	1.0	0.034	0.009	17.4	11.8	0.20	—

* Al and/or Ca are added depending on the occasions.

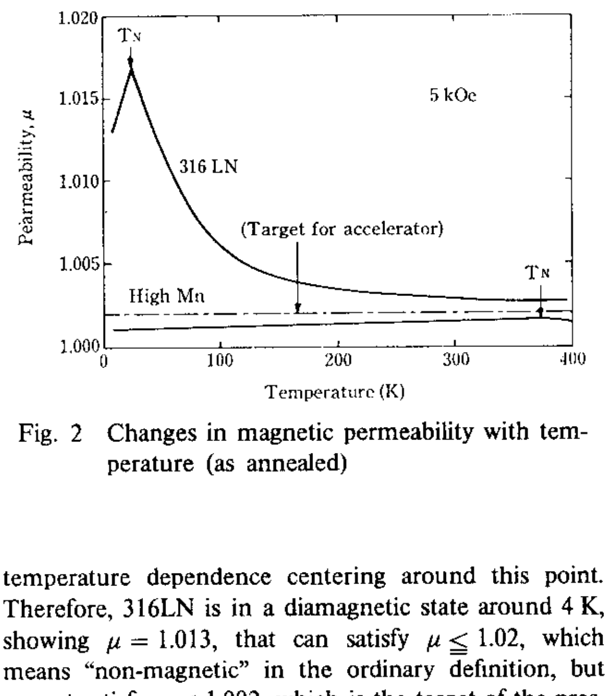

Fig. 2 Changes in magnetic permeability with temperature (as annealed)

temperature dependence centering around this point. Therefore, 316LN is in a diamagnetic state around 4 K, showing $\mu = 1.013$, that can satisfy $\mu \leqq 1.02$, which means "non-magnetic" in the ordinary definition, but cannot satisfy $\mu \leqq 1.002$, which is the target of the present research. On the other hand, T_N also exists in the newly developed Mn steel, but since its value is 375 K, which is above room temperature, the degree of magnetization in the Mn steel is far decreased than that of 316LN. Thus the Mn steel satisfies the conditions of $\mu \leqq 1.002$ even at the T_N temperature. In short, the high-Mn steel shows $\mu \leqq 1.002$ within the measured temperature range of 4 to 400 K, and its temperature dependence is very small.

The supporting collar material is required to have high strength (yield stress at RT: $\sigma_y \geqq 620$ MPa) in order to cope with the localization of electromagnetic force and prestress. To achieve this, temper cold rolling is adopted, where the strain dependence of specific magnetic permeability poses a problem. The measurements of magnetic permeability in the high-Mn steel compared with 316LN are shown in **Fig. 3**, which indicates a significant difference between the two types of steels. While the high-Mn steel shows no rise in magnetic permeability (at 4 K) even with a cold-rolling deformation of 30% or more, 316LN generates an abrupt rise in μ by applying deformation.

The above-mentioned tests have proved that the newly developed high-Mn steel, which has been made by adding N and V to the low C-high Mn matrix, has only a small value of magnetic permeability as well as minimum temperature- and strain-dependence, and poses no magnetic problem, even if its strength is controlled by temper rolling. It has also been found that its magnetic permeability fluctuations of inter lots sufficiently fall within 0.0005.

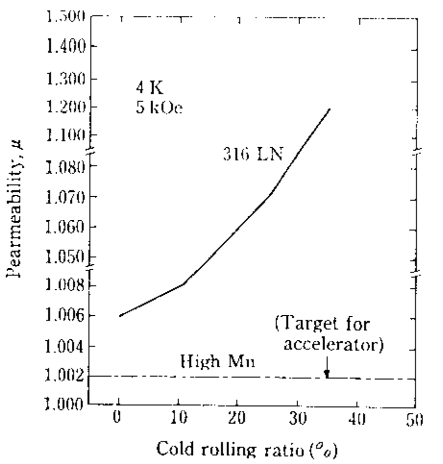

Fig. 3 Changes in magnetic permeability with cold working

3.2 Mechanical Strength and Toughness

Both the electromagnetic force, which is generated by a magnetic field of 6.6 tesla during the operation of the superconducting magnet, and pre-stress for suppressing the contraction or expansion between the superconducting cable covered with the heat-insulating material and supporting collar are liable to localize. To cope with this, it is necessary to sufficiently strengthen yield stress σ_y. Namely, $\sigma_{y(4\,K)} \geqq 1\,200$ MPa and $\sigma_{y(RT)} \geqq 620$ MPa are set up as targets, and they must be achieved while specific magnetic permeability μ (4 K or RT) $\leqq 1.002$ and its fluctuation (dispersion) $\Delta\mu \leqq 0.0005$ are satisfied. It has been found that the yield stresses of this alloy, namely, $\sigma_{y(4\,K)}$ and $\sigma_{y(RT)}$ can achieve their targets by the addition of a solute element, N, and by temper cold rolling, respectively.[4]

Effects of N addition on yield stress $\sigma_{y(4\,K)}$ and elongation (λ) of the high-Mn steel having a composition of 0.2%C-28%Mn-0.1%V, when the steel is subjected to cryogenic uniaxial tensile deformation, are shown in **Fig. 4**, which indicates that yield stress at 4 K rises with N addition. This was also observed for some austenitic steels,[5] and suggests that it is generally applicable to austenitic steels. On the other hand, cryogenic ductility will not change by N addition up to slightly above 0.15%, but when N is further added, the cryogenic ductility suddenly drops. Furthermore, any large amount of N addition will not only be a burden on the melting and refining process, but also a generation of problems such as blowholes during welding operations. It is advisable, therefore, to set the limit of N addition to 0.15% as shown in Table 1.

Yield stress at room temporature, $\sigma_{y(RT)}$, in the as-annealed state of high-Mn non-magnetic steel, whose composition is shown in Table 1, is about 350 MPa as shown in **Fig. 5**, which is slightly larger than $\sigma_{y(RT)}$ of 316LN austenitic stainless steel. This yield stress must be upgraded at least to 410 MPa (60 000 psi) or to a target value of 620 MPa (90 000 psi). It is conceivable that in Fe-Mn-based alloys the above requirement can be achieved by precipitation hardening using carbides with V, Nb and Ti[6], but in the present alloy system, a low-C-based alloy is adopted taking into consideration hot workability, weldability and cryogenic toughness, and, as mentioned above, the magnetization (magnetic permeability) is almost strain-independent because of high magnetic stability. For this reason, the authors have examined the possibility of controlling the yield stress by temper cold rolling at the final process of sheet manufacturing. According to Fig. 5, yield stresses of $\sigma_{y(RT)} > 410$ MPa and $\sigma_{y(4\,K)} > 620$ MPa can be attained by temper rolling of about 5% and about 15%, respectively. As shown in Fig. 3, there is absolutely no possibility of an increase in specific magnetic permeability.

Incidentally, superconducting cables for accelerator superconducting magnets currently working or in a

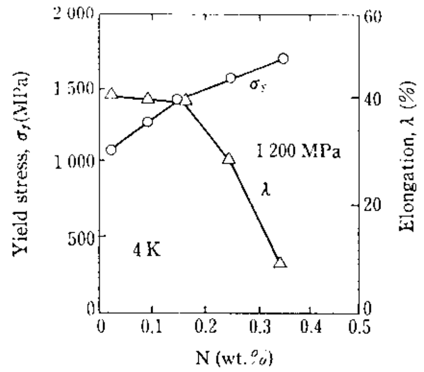

Fig. 4 Effects of N on tensile properties of high Mn steel at 4 K (as annealed)

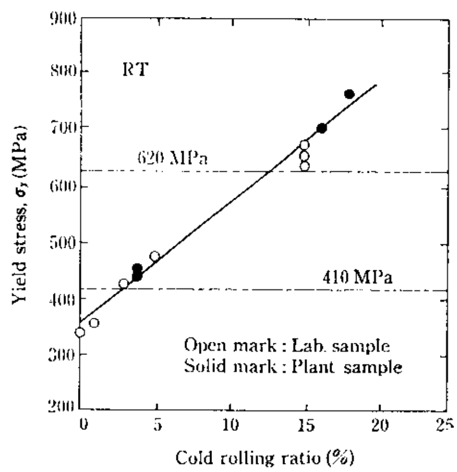

Fig. 5 Changes in yield stress σ_y at RT with cold temper-rolling

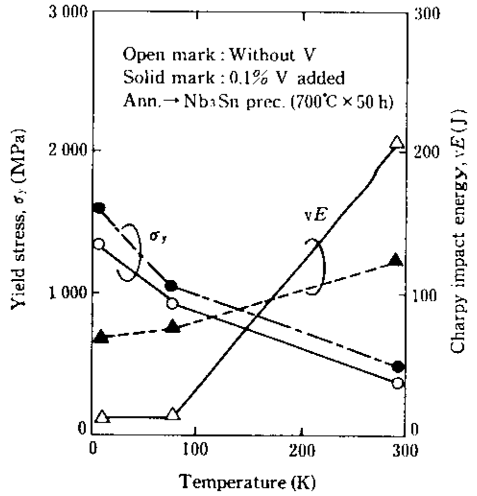

Fig. 6 Temperature dependence of yield stress σ_y and Charpy impact energy $_vE$ of Nb_3Sn heat treated high Mn steel with and without V

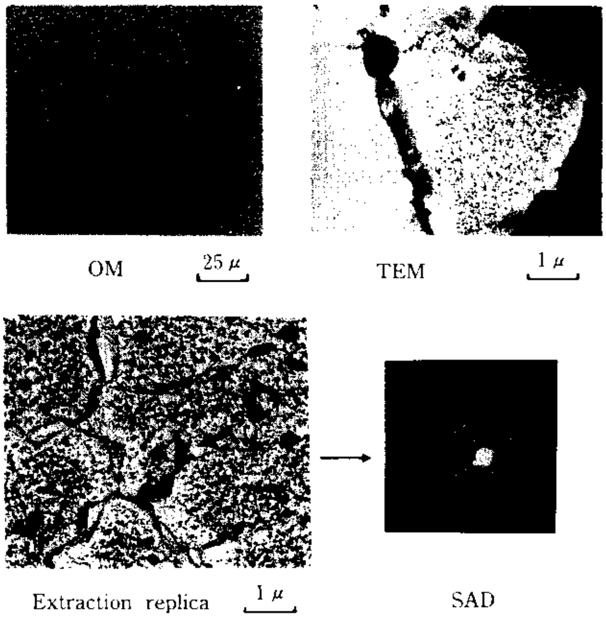

Photo 2 Optical and electron micrographs of V containing Mn steel subjected to Nb_3Sn precipitation heat treatment

planning stage are of the alloy-type NbTi-based material, but in the future, there is a possibility of using Nb_3Sn type inter-metallic compounds in order to attain higher performance and a more compact size. If the "Wind and React Method" is available, the application of the Nb_3Sn superconductors can be expanded. However, the "React" treatment of Nb_3Sn (in the present research, strict conditions were adopted by using a precipitation heat treatment of 700°C × 50 h) caused a significant drop in cryogenic toughness, when V was not added, in the high-Mn steel, as in the case of 316LN, as shown in **Fig. 6**. It was found that at 4 K, Charpy impact absorption energy $_vE$ dropped to practically nil. The cause of these drops may be attributable to embrittlement of the grain boundary by the precipitation and agglomeration of Cr carbides along grain boundaries, as was observed in austenitic stainless steels.[7,8] Then, it can be seen in Fig. 6 that with an addition of V to the high-Mn steel, the solid solution hardening given by V not only increases σ_y to some extent, but also largely inhibits the cryogenic temperature deterioration of $_vE$, demonstrating the values as high as about 80J even at 4 K. The cause of this is morphologically examined in **Photo 2**, which indicates the optical and transmission electron microscopic structures as well as an extraction replica and its electron beam diffraction image. Numerous numbers of precipitates observed in the crystalline grains are found, with the aid of selected area diffraction (SAD), to be V carbonitrides formed during the precipitation treatment. This suggests that such micro-precipitates suppress the grain boundary segregation and agglomeration of Cr carbides, thereby preventing the drop of grain boundary resistance against deformation at the cryogenic temperature and also limiting the deterioration of ductile toughness. Consequently, there is a strong possibility for the V-addition high-Mn non-magnetic steel to be applied to the case in which the Nb_3Sn superconductor is used by the "Wind and React Method."

3.3 Application Characteristics

Since the collar support member of the accelerator superconducting magnet becomes a laminated structure made of steel sheets of about 1.5 mm in thickness, what are considered important as fabricabilities of non-magnetic supporting materials are stampability and spot-weldability (rusting and corrosion resistances will not be a problem, if rust and corrosion are prevented during the magnet manufacturing process, because the actual operation environment of the magnet consists of vacuum at cryogenic temperature). Therefore, these two kinds of required performance of the newly developed high-Mn steel were investigated by comparing them with those of 316LN stainless steel.

Since stampability is governed by strength and work-hardening characteristics and the newly developed material is hardened by temper rolling at the final process, its stampability was compared with that of 316LN in the ordinary as-annealed state. The machine used in the test is a 10-t HP hydraulic stamping machine and **Table 2** shows the testing conditions. For the stamping tool material, SKD11 was used, and blank diameters were of three kinds. The clearances were of two kinds of 0.08 mm and 0.14 mm for each blank diameter. No lubricant was used. In the present stamping test, where it is difficult to examine the tool life or the deteriora-

Table 2 Experimental stamping conditions

Sample	① High Mn ② 316LN
Sample thickness	1.5 mm
Hydraulic machine	10 t HP
Tool material	SKD 11
Blank diameter	① 12 mmφ ② 18 mmφ ③ 24 mmφ
Clearance	① 0.08 mm ② 0.14 mm

* No lubricant was used

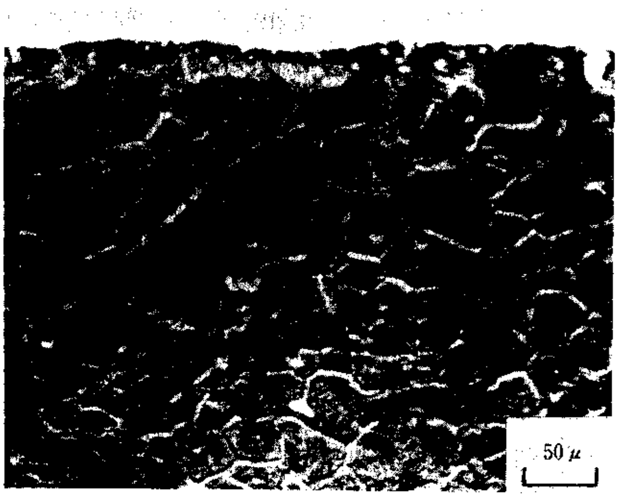

Photo 3 Scanning electron micrographs of high Mn steel after stamping

Table 3 Measured height of burr after stamping (μm)

Blank \ Clearance	High Mn		316LN	
	0.08 mm	0.14 mm	0.08 mm	0.14 mm
12 mmφ	90	89	98	94
18 mmφ	78	76	82	76
24 mmφ	72	62	78	81

Table 4 Experimental conditions of spot welding

Voltage	0.5~1.3 V
Current	1.6~7.3 kA
Load	600 kg
Welding cycle	5, 10 and 20
Electrode (chip)	Cu; dome-type, $d=5\sqrt{t}$
Mechanical test	Cross type tensile test

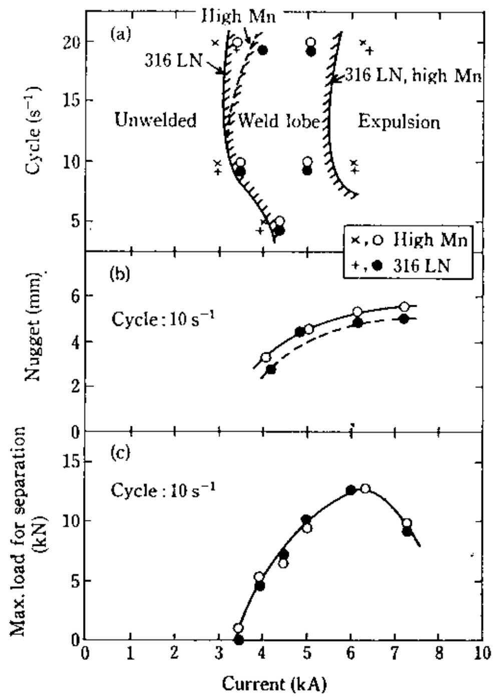

Fig. 7 Results of spot welding test

tion degree of the tool, the degree of burrs after stamping and stamping load were measured. **Photo 3** shows the appearance of burrs generated during the test, which were observed on a scanning electron microscope. **Table 3** shows the measurements of individual burr heights. The Table shows that the burr height becomes lower, as the blank diameter becomes larger, not being affected by the clearance. Further, under any conditions, no sign has been observed that the high-Mn steel is more liable to develop burrs than 316LN. In addition, the maximum stamping load, when the blank diameter is 24 mmφ and the clearance is 0.08 mm, amounts to 240 kg for 316LN and to 210 kg for the high-Mn steel. This means that although the high-Mn steel is far stronger than 316LN ($\sigma_y > 620$ MPa for the former and $\sigma_y \approx 250$ MPa for the latter), the high-Mn steel shows slightly lower burr heights and smaller stamping loads. It is considered therefore that the high-Mn steel is not inferior to austenitic stainless steel at least in stampability.

The spot weldability of the high-Mn steel has been investigated using a spot-welding machine. The welding test conditions are shown in **Table 4**. With the load kept at a constant value of 600 kg, and the amperage (voltage) and welding cycle as parameters, the weld lobe, nugget width and load for separation (by the tensile test on the cross type laminated specimens) were obtained. The test results are arranged in **Fig. 7**. There are no virtual differences between 316LN and the high-Mn steel in the weld lobe position, amount of expulsion, nugget width and load for separation (the nugget height rather tends to be slightly larger for the high-Mn steel). In the external appearance of specimens after spot welding, there was no particular difference between the two types of steel. Namely, the high-Mn steel is not considered to be inferior in spot-weldability to the austenitic stainless steel. In addition, the specific magnetic permeability at the spot-welded zone of the high-Mn steel is maintained at 1.002 or below, indicating that magnetism of this steel will not be deteriorated at all.[9)]

4 Conclusions

The results of research and development of a cryogenic non-magnetic supporting collar material for large super-conducting magnets for use inaccelerators are summarized as follows:

(1) Through the composition design (low C-high Mn-N-V) of high-Mn austenitic steel, the authors achieved a cryogenic specific magnetic permeability of 1.002 or below with its fluctuation of 0.0005 or below. These results are desirable for curbing magnetic field fluctuations.

(2) The high-Mn steel shows little change in the specific magnetic permeability under an applied strain of 30% or above and with temperature change of between 4 K and 400 K, and demonstrates high magnetic stability. This is desirable for designing material strengths and ensuring stable operations of magnets.

(3) Through N and V additions and temper rolling, the yield strength of the high-Mn steel has been improved to 629 MPa at room temperature and to 1 200 MPa at 4 K. This is desirable against the loads of electromagnetic force and pre-stress.

(4) An addition of V into this steel has made it possible to suppress the deterioration of cryogenic ductile toughness after precipitation heat treatment in the "Wind and React Method" of the Nb_3SN superconductor. This is desirable for the future use of Nb_3Sn in place of NbTi.

(5) In terms of stampability and spot weldability which are significant properties in practical manufacture of magnets, the high Mn steel has been found to have virtually the same level of performance as that of 316 LN austenitic stainless steel.

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