

# A New Welding Material for Improved resistance to Ductility Dip Cracking

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## Abstract

Nickel-Chromium-Iron alloys and welding products have been used throughout the life of the nuclear welding industry. From the beginning, Welding Electrode 182 and Filler Metal 82 were used to weld alloy 600 until they were found to be susceptible to primary water stress-corrosion-cracking (PWSCC). This brought about the need for a 30% Cr-containing alloy 690 along with Welding Electrode 152 and Filler Metal 52. These materials were resistant to PWSCC, but the welding products were found to be susceptible to ductility dip cracking (DDC). Because DDC is a solid state cracking phenomenon that may occur in highly restrained austenitic welds, most of the existing tests were ineffective for measuring DDC. The Ohio State University welding group introduced the Strain-to-Fracture (STF) test to measure the tendency for DDC. A number of nuclear welding materials have been subjected to STF testing and a new nickel alloy material with 30% chromium, INCONEL<sup>®</sup> FM52MSS, has shown substantially improved DDC cracking resistance when measured with the STF test. This paper discusses the development and performance of the welding material and the results of STF test of this new alloy.

## Introduction

From the initiation of the commercial nuclear power generation industry, INCONEL<sup>®</sup> alloy 600 and INCONEL<sup>®</sup> Welding Electrode 182 (ENiCrFe-3) and Filler Metal 82 (ERNiCr-3) were chosen to resist the corrosion environment in nuclear reactors and steam generating equipment. After several years of worldwide operating experience, these materials were found to be susceptible to primary water stress corrosion cracking (PWSCC) due to their insufficient chromium contents [1-4]. Therefore, the alloy 600 and welding materials 182 and 82 have largely been replaced with 30% chromium-containing materials known as INCONEL<sup>®</sup> alloy 690 and INCONEL<sup>®</sup> Filler Metal 52 and Welding Electrode 152 in USA and other countries. These materials

have been shown to be free from (PWSCC) cracking in operating reactors for over 15 years [1].

However, fabricators have found and identified a phenomenon known as ductility-dip cracking (DDC) that may occur during weld fabrication which has been found to be associated with austenitic materials of several types such as nickel-base, Ni-Cu alloys, Cu alloys, and stainless steels. Early ductility-dip cracking (DDC) that formed in weld metals was usually small, and was often referred to as a 'micro-fissuring'. Although this type of cracking (DDC) was identified as early as 1961 by Rhines and Wray [5], this term micro-fissuring was indiscriminately applied to both solidification cracking and to DDC until the early 1990's.

DDC is a solid-state, elevated temperature phenomenon that has been observed in thick-section, multipass austenitic stainless steel and nickel-based alloy weld metals characterized by large grain size and high restraint. An example is shown in Figure 1. The mechanism has been postulated to be the result of "ductility exhaustion" shown in Figure 2 along the grain boundary with grain boundary sliding and the relative orientation of a grain boundary to an applied strain increasing susceptibility to DDC. Although the occurrence of DDC is sometimes unnoticed, in applications where there is low defect tolerance, such as nuclear fabrication, its minimization is highly desirable.

INCONEL<sup>®</sup> Filler Metal 52M (ERNiCrFe-7A) has reported the improvements of DDC resistance over INCONEL<sup>®</sup> Filler Metal 52, however, the applications involving overlays (PWOL, SWOL, etc.) are insufficiently demanding to highlight the severity of cracking that can occur with higher restraints and heavier section welds[6]. In fact, the advent of much improved weld bead cleanliness presented by 52MS had a greater impact on product acceptance than improvements in DDC resistance. Recently a new generation welding material of the 52 family, INCONEL<sup>®</sup> Filler Metal 52MSS, was invented and patented by Special Metals Welding Products

Company and AREVA. This new alloy contains about 2.5 wt.% Nb and 4.0 wt.% Mo, which have been shown to improve DCC resistance dramatically. This paper will present the substantially improved DCC resistance of this alloy and the function of Nb and Mo on DCC resistance.



Figure 1, Ductility dip cracking (DDC) of trailing, solidified portion of linear vareststraint test.

### Materials and Sample Preparation

Six high-Cr weld metals (denoted as 3W-1, 3W-2, 3W-3, 3W-4, 52MSS (1), and (2)) with varying contents of Nb and Mo were used in this study. The chemical compositions of these alloys are presented in Table 1. Usually INCONEL® Filler metal 52MSS contains ~30% Cr, ~2.5% Nb and ~4.0% Mo and other elements. Other different types of Ni-base filler metals are given in Table 1 for comparison.

Multipass butt joint plates were made by the automated gas tungsten arc welding (GTAW) and gas metal arc welding (GMAW) processes. The base metal used for these joints was alloy 690. Dog bone-like samples, as shown in Figure 3, were sectioned transversely from the slotted butt joint plates and ground to the final dimension. Then, a GTAW spot weld was made at the middle of the pre-deposited weld on both sides, as presented in Figure 3. The circular geometry of this spot gives

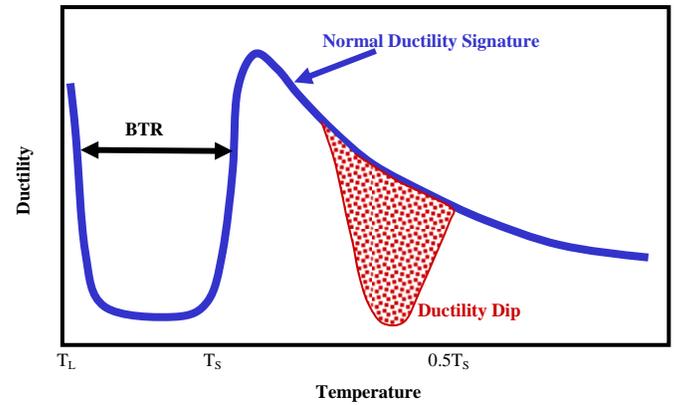


Figure 2. Schematic that represents the mechanism of ductility-dip cracking.

a radial distribution of grain boundaries so that cracking resulting from the axial strain during the STF test will occur along the most susceptibly oriented grain boundaries. Complete details of the STF test are given in Reference 7.

The tested samples were then evaluated for cracks using a stereo microscope up to 30X magnification. The degree of DDC in a given sample is determined by counting the number of cracks in the elongated spot weld of both sides of the sample, then dividing by two. Only cracks that were distinguishable at up to 30X magnification were included in the count. Subsequently, the STF tested samples were prepared for metallographic observation by grinding, polishing and then electrolytic etching in a 10% chromic acid solution at 1.5-2V for 30 s. Scanning electron microscopy (SEM) and EDS analysis was performed on a Sirion SEM/FEG and Phillips XL-30 ESEM/FEG at 15kV.

Table 1. Chemical compositions of different nickel base filler metals (wt.%)

Element	FM82	FM52	FM52M	3W-2	3W-3	3W-4	3W-1	52MSS (1)	52MSS (2)	68HP	69HP
C	0.031	0.026	0.02	0.026	0.019	0.012	0.02	0.026	0.024	0.029	0.005
Mn	2.97	0.24	0.80	0.05	0.97	2.64	0.18	0.19	0.79	0.90	1.21
Ni	72.34	58.82	59.54	56.62	59.73	58.31	61.79	54.67	53.46	58.81	57.0
Cr	20.57	28.91	30.06	29.0	29.24	26.8	29.11	29.92	30.34	29.94	30.0
Fe	0.95	10.53	8.22	8.35	6.30	9.04	3.24	8.31	8.18	8.80	9.15
Nb	2.5	0.03	0.83	2.46	2.11	2.53	2.19	2.57	2.49	<0.01	1.83
Mo	-	0.04	0.01	1.87	0.75	<0.01	3.04	3.83	4.01	<0.01	<0.01
S	0.002	<0.001	0.001	<0.001	0.001	0.012	0.001	0.0013	0.0014	0.001	0.001
P	0.004	0.004	0.003	<0.003	<0.003	0.006	0.001	0.0001	0.009	<0.003	0.004
Ti	0.32	0.55	0.224	0.34	0.32	0.13	0.231	0.193	0.188	0.68	0.39
Al	-	0.66	0.11	0.24	0.22	0.10	0.069	0.07	0.218	0.75	0.20
Si	0.16	0.15	0.09	0.08	0.08	0.39	0.115	0.119	0.21	0.13	0.11
Cu	0.01	0.02	0.02	0.06	0.04	0.01	0.013	0.059	0.06	<0.01	<0.01
Mo+Nb	2.5	0.07	0.84	4.33	2.86	2.53	5.23	6.4	6.5	<0.02	1.83

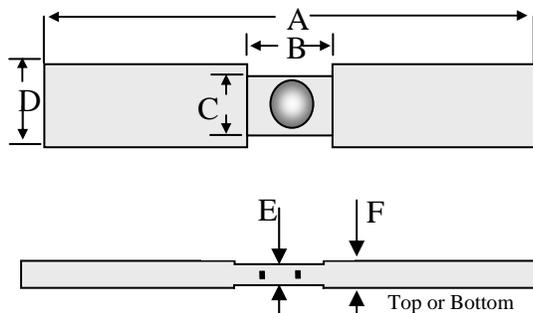


Figure 3. Schematic illustration of strain-to-fracture sample.  $A = 12.7 \text{ cm}$  (5 inches),  $B = 19 \text{ mm}$  (0.75 inch),  $C = 15.3 \text{ mm}$  (0.6 inch),  $D = 19 \text{ mm}$  (0.75 inch),  $E = \text{Nominally } 5.6 \text{ mm}$  (0.22 inch),  $F = 6.4 \text{ mm}$  (0.25 inch).

## Results and Discussions

STF test results for filler metal 52MSS are presented in Figure 4. The numbers in parenthesis represent the number of cracks found on both sides of the sample divided by two. The black numbers represent the DDC results of an early experimental heat 52MSS then identified as 52X-H. The black solid line represents the approximate threshold strain for cracking for the initial experimental heat of 52MSS.

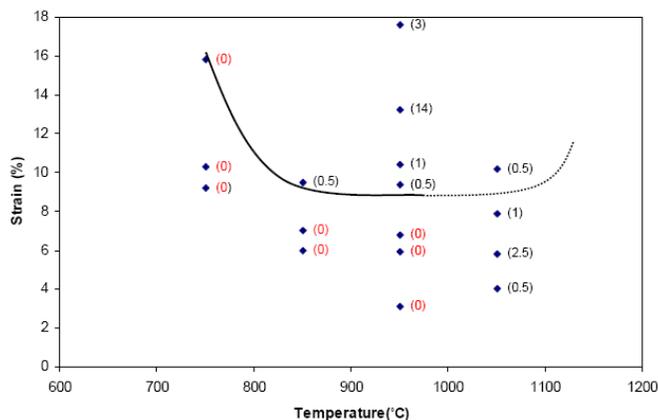


Figure 4. Initial STF results for Filler Metal 52MSS (then called 52X-H).

The STF results for Filler Metal 52MSS are compared to the STF data published for Filler Metal 52 and Filler Metal 82, as shown in Figure 5[6-7]. The results indicate that the DDC resistance of the initial heat of Filler Metal 52MSS is much better than Filler Metal 52, another 30% Cr nickel-base alloy, and even superior to Filler metal 82, which is known to be moderately resistant to DDC under high-restraint welding condition. Because the most susceptible temperature for DDC to occur in 30% Cr Nickel-base welding alloys is 950°C, preliminary screening is often performed at this temperature.

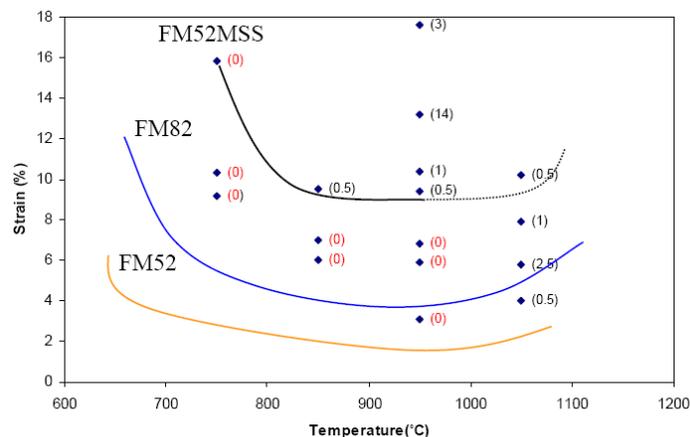


Figure 5. Comparison of the Filler Metal 52MSS threshold strain for cracking to the STF results of Filler Metal 52 and 82.

A comparison of the STF results of different welding filler metals tested at 950°C is provided in Figure 6. The horizontal bar in the column above each filler metal represents the threshold strain for cracking for that material while the numbers present the number of cracks that were present in the sample as a function of the strain. The temperature of 950°C was selected for STF screening testing due to the maximum ductility-dip at this temperature illustrated in Fig. 2. In addition, experience has indicated that the cracking response at 950°C correlates well with actual fabrication behavior and provides the most discerning data.

The STF results of three different heats of FM52M and three different heats of FM52MSS were shown in the Fig 6. FM52M-1 is an old heat sample tested two years ago. But recently two 52M samples (FM52M-2 and FM52M-3) after tightly controlling the minor elements and applying a special clean procedure show very good DDC resistance, FM52M-2 is close to FM82 performance while FM52M-3 is superior to FM 82. The threshold strain for cracking for FM52MSS is between 8-16% at 950°C. These data are generated from testing 52X-H, FM52MSS-1, and FM52MSS-2 shown in Table 1 which contain 52MSS prescribed amounts of Nb and Mo. There are no cracks for the FM52MSS-1 sample at an applied strain of 15%, and no cracks for the FM52MSS-2 sample at an applied strain of 16%. No STF tests were done beyond these strains.

The data presented in the Fig. 7 provides the trend of the threshold strain for cracking with the content of Nb and Mo in experimental welds. It is clear that with increasing amounts of Mo in the filler metal when Nb is between 2% and 3%, the threshold strain for cracking increases. It is indicated that the 52MSS family containing 5-6.5% Nb+Mo when %Nb is at least 2%, exhibit the highest threshold strains and the best resistance to DDC cracking.

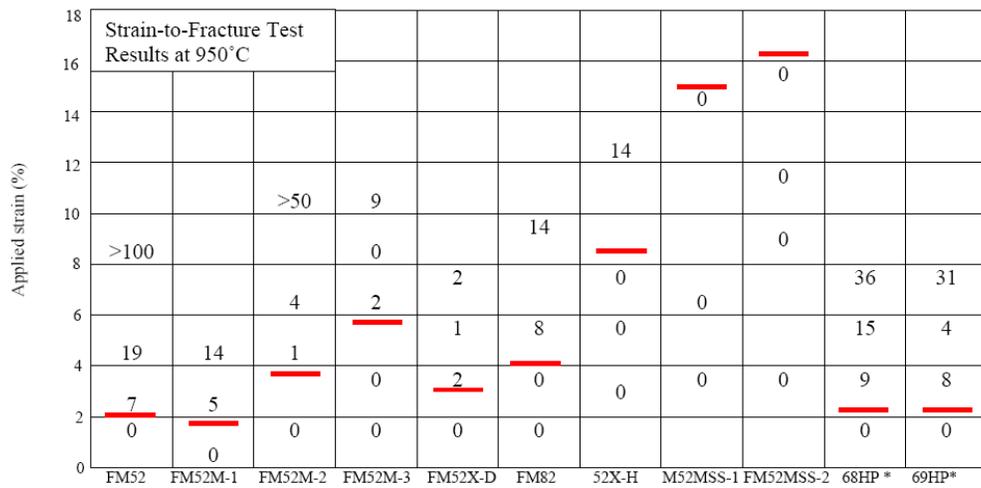


Figure 6. Strain-to-fracture test results for different nickel-base weld metals tested at 950°C. Some data from reference 7.

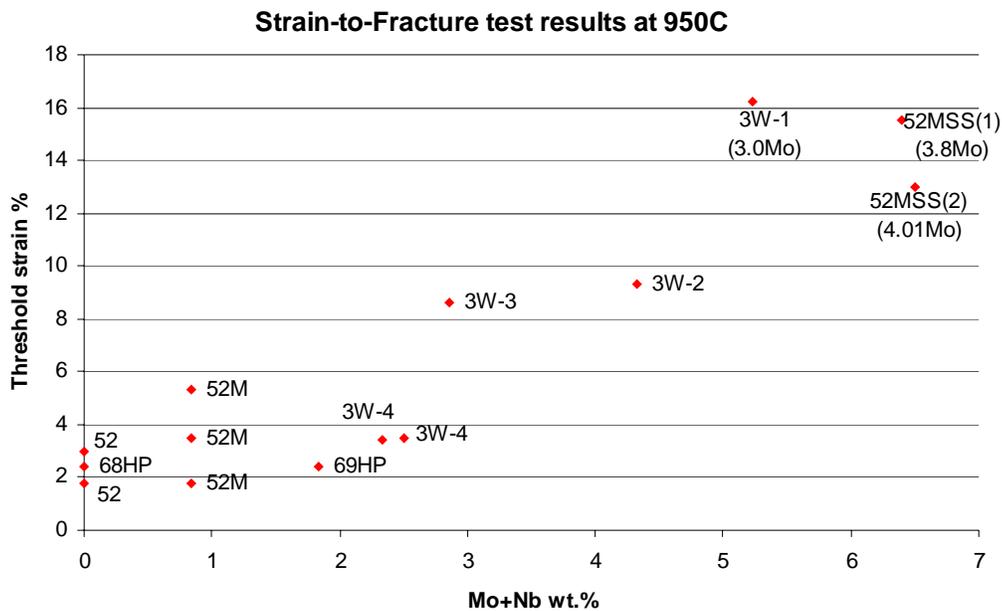


Figure 7. The relationship between threshold strain for cracking and the content of Ni and Mo. The STF results tested at 950°C. Note that alloy 3W-4 and data points to the left contain no Mo while 3W-3 and alloys to the right have Mo additions and show much improved STF performance.

In order to investigate the influence of Nb and Mo on the DDC resistance, microstructural examination of FM52M and FM52MSS was conducted by OSU researchers after STF testing. FM52M contains approximately 0.8% Nb and no Mo, boundaries are extremely serpentine. The straight grain boundaries of FM52M were populated with small, Cr-rich  $M_{23}C_6$  carbides as shown in Fig 9. These carbides form in the solid-state as the weld metal cools to room temperature and were believed to have no effect on boundary pinning. The SEM image of FM52MSS weld metal shown in Fig. 10 indicates that the Nb-rich M(C,N) precipitates are spread

while FM52MSS has a combined total of 6.5% Mo+Nb. Figure 8 show the patterns of migrated grain boundaries using electron backscattered diffraction (EBSD). It is clear that the FM52M boundaries are generally straight, while the 52MSS throughout the weld metal interdendritically instead of only in the grain boundaries. These well-distributed precipitates form at the end of solidification and are effective at pinning the migrating grain boundaries, resulting in serpentine grain boundaries that create interlocking grains. This structure is much more effective at resisting grain boundary sliding and thus greatly improves DDC resistance.

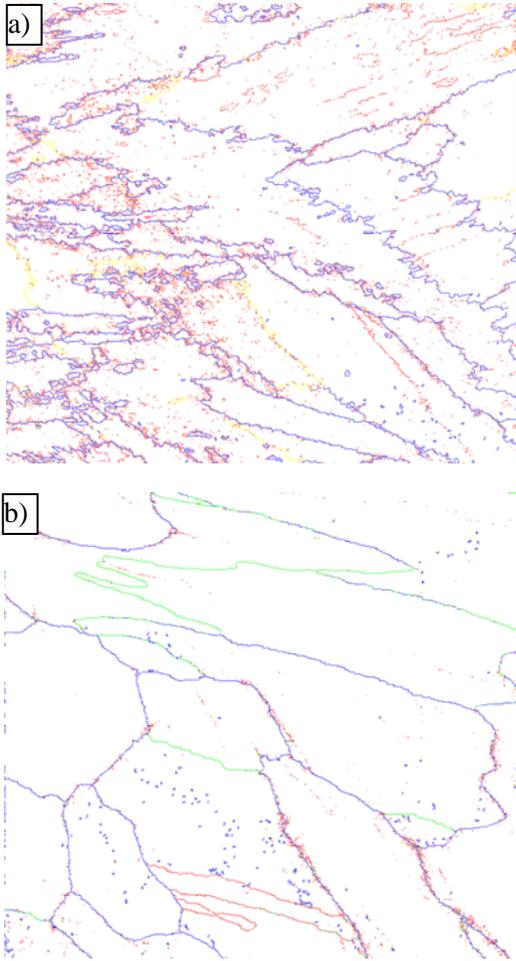


Figure 8. Electron backscattered diffraction (EBSD) patterns showing (a) serpentine migrated grain boundaries in FM52MSS and (b) the nearly straight grain boundaries of FM52M.

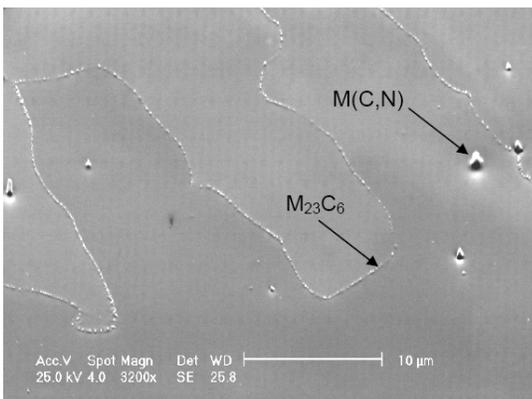


Figure 9. Undesirable long, straight grain boundaries with nearly continuous  $M_{23}C_6$  carbides in FM 52M.

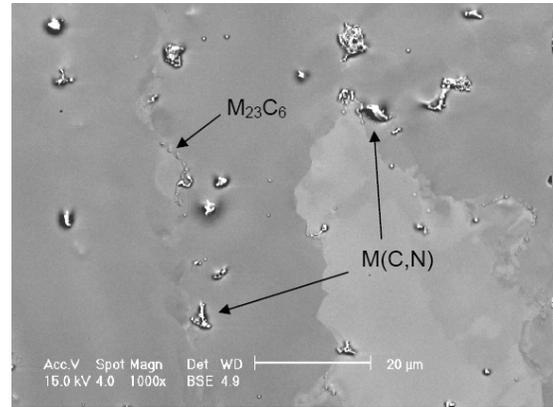


Figure 10. Backscattered electron SEM image showing visible  $M(C,N)$  precipitates and  $(Nb,Ni)$ -rich phases in the interdendritic regions and  $M_{23}C_6$  carbides along a few of the grain boundaries. Noticeable grain boundary pinning by the large intragranular precipitates is still observed.

## Summary

The current status of Nickel based alloys for nuclear construction is that 30% chromium-containing nickel-base alloys and welding products are necessary for primary water stress corrosion cracking (PWSCC) resistance. These materials have sufficient resistance to PWSCC, but the weld metals suffer from susceptibility to DDC cracking due to the tendency for long, straight grain boundaries during solidification and cool down. INCONEL<sup>®</sup> Filler Metal 52M provides reasonable resistance to DDC during fabrication and good resistance to PWSCC in nuclear service. But the newest 30% chromium-containing nickel alloy welding product, INCONEL<sup>®</sup> Filler Metal 52MSS, has been shown to provide outstanding DCC resistance and has been found in other research[8] to provide the same excellent PWSCC resistance as INCONEL<sup>®</sup> Filler Metal 52M.

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