

# Ductility Dip Cracking of Ni-base Filler Metals—Insight into the Mechanism

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## Introduction

Ductility dip cracking (DDC) is an intermediate temperature, solid-state, intergranular cracking phenomenon, which has been reported in several alloys systems such as stainless steels<sup>1,2</sup>, Ni-base alloys<sup>3-8</sup>, aluminum alloys, copper alloys, and titanium alloys<sup>9,10</sup>. This cracking phenomenon occurs during high temperature processing such as hot rolling, forging and welding. Specifically, in the case of welding, cracking is normally manifested when highly restrained welds are performed in these susceptible materials. Several factors have been reported to influence DDC: impurity and interstitial element segregation to grain boundaries (GB), grain size, GB orientation related to applied stress, grain boundary “tortuosity”, welding parameters, and strain rate. However, the effect of each of the factors is not clear and the DDC mechanism is still not fully understood, which makes eliminating DDC extremely difficult.

This work is an update of the continuing effort at The Ohio State University to develop test procedures that quantify susceptibility and identify the mechanisms associated with DDC. The strain-to-fracture (STF) test has previously been described<sup>1</sup> as an effective method to evaluate susceptibility to DDC. In this paper, microstructural characterization conducted on STF samples of two Ni-base filler metals is presented and insight into the DDC mechanism is provided.

## Experimental Procedures

Multi-pass, cold wire, GTAW welds performed with Filler Metal 82 (FM-82) and Filler Metal 52 (FM-52) were tested using the STF technique in a Gleeble™ 1500 system<sup>8</sup>. In this test, a sample (Fig. 1) is heated to the test temperature and held at that temperature for a short time, and then subjected to a preset strain at a certain rate. An example of the test conditions is shown in Fig. 2. Finally the DDC susceptibility is measured by counting the number of cracks on the sample surface using a stereoscopic microscope at up to 70x magnification.

The chemical compositions of the filler metals are shown in Table 1. Optical and electron (SEM and TEM) microscopy, along with chemical microanalysis (EDS) and electron-backscattered diffraction (EBSD) were used for microstructure analysis.

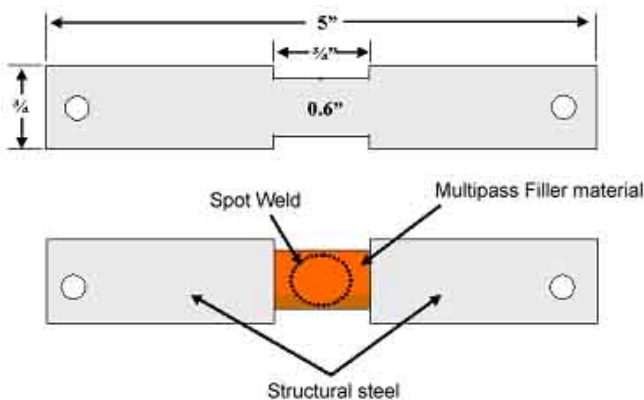


Figure 1: Strain-to-fracture test sample<sup>8</sup>.

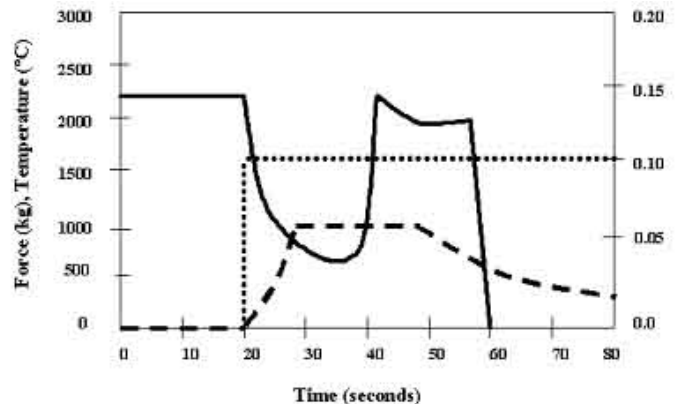


Figure 2: Schematic presentation of the STF test parameters<sup>8</sup>.

**Table 1:** Chemical composition (wt%) of the filler metals

Alloy	%C	%Mn	%Fe	%S	%Si	%Cu	%Al	%Ti	%Cr	%Nb+Ta	%Mo	%P	%Co	%Ni
FM-82*	0.04 0	2.86	1.18	0.00 1	0.12	0.09	-	0.37	20.1	2.3	-	0.00 7	0.05	72.7 5
FM-52**	0.02 6	0.25	8.88	0.00 4	0.17	0.01 1	0.71	0.50	29.0 9	0.02	0.05	0.00 4	-	60.1 2

\* Filler Metal 82 heat YN6830

\*\* Filler Metal 52 heat NX9277

### Results and Discussion

Based on the STF data collected by Collins et al.<sup>8,11</sup>, 3-D plots showing the DDC susceptibility were developed for these alloys, as shown in Fig. 3. From these plots, the ductility trough extends over a large temperature range (approx. from 625 to 1200 °C) for both alloys. The alloy FM 52 is more susceptible to DDC because the cracking occurs at lower applied strains. FM-82 begins to consistently form cracks with about 5% of strain. Conversely, the FM-52 starts cracking when approaching 2.5% of strain. In addition, the number of cracks in FM-52 increases rapidly with an increase in strain within the DDC range.

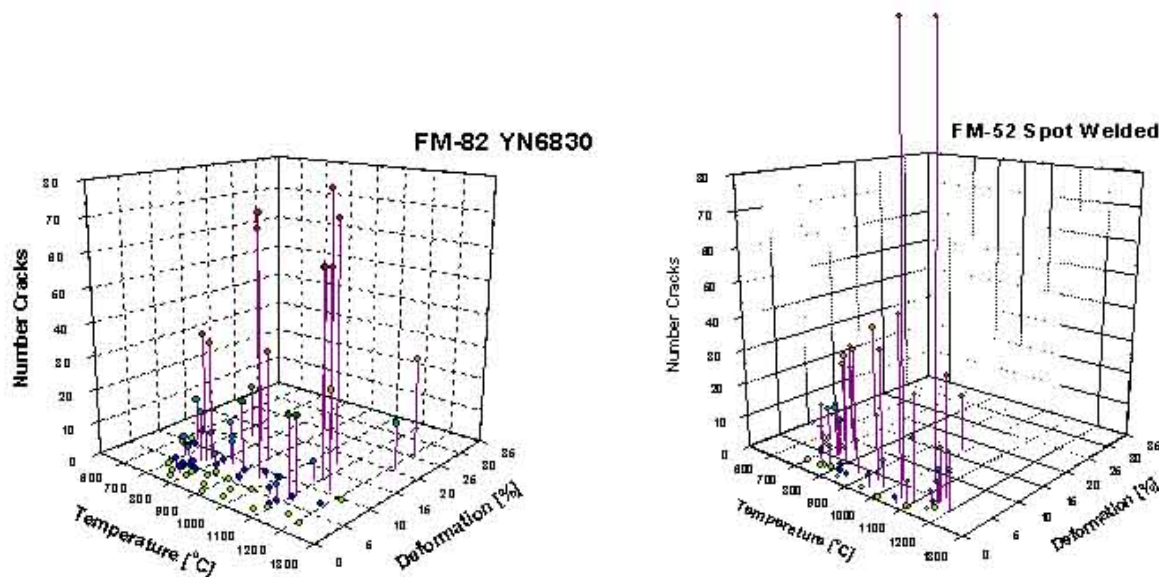


Figure 3: 3-D DDC susceptibility curves for multipass weld deposits of FM 82 and FM 52.

The most important microstructural difference between the filler metal deposits was the nature, distribution and fraction of precipitates. The precipitates in both weld metals were crystallographically and chemically characterized on the SEM and TEM by EBSD, XEDS, SAD, and CBED. FM-82 exhibited a considerable amount of precipitates distributed in three different populations. The first population was discernible in the optical microscope and consisted of an extensive, uniform distribution of large (about 1  $\mu\text{m}$ ) eutectic (Nb,Ti)C type carbides. These precipitates were intergranular and intragranular. The second population was formed by medium sized (100 nm) (Nb,Ti)C carbides, which were predominately intergranular. Some intragranular colonies were also observed in the interdendritic regions. The third population of precipitates was formed by small (10 nm) intergranular particles that coexist with the intragranular medium size carbides. The FM-52 presented three populations of precipitates as well, but of different nature and with different distributions relative to FM-82. The first type of precipitate in FM-52 consisted of large cuboidal (1-5  $\mu\text{m}$ ) TiN particles, mostly intragranular, which were easily observable using the optical microscope. However, these precipitates were unevenly distributed within the microstructure. The large size of these nitrides and the presence of much larger ones in the filler

wire strongly suggest that these high melting temperature nitrides were transferred from the filler wire and partially dissolved during the welding process. Thus, the uneven distribution of these large nitrides through the weld metal should be related with the liquid metal flow patterns and the solidification process. The second group of precipitates consisted of abundant, medium sized (10-50 nm)  $M_{23}C_6$  intergranular carbides. The third group of precipitates observed in FM-52 was composed of evenly distributed, very small (about 10 nm) precipitates, which are suspected to be nitrides or carbo-nitrides. SEM pictures of precipitates along migrated grain boundaries in both materials are shown in Figure 4.

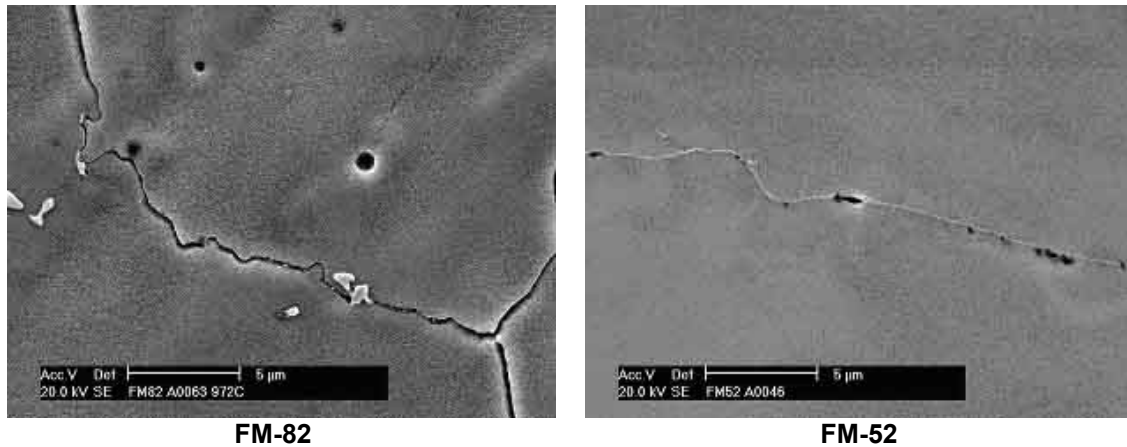


Figure 4: Secondary electron SEM image of STF samples of FM-82 and FM-52.

One of the most important microstructural differences between FM-82 and FM-52 weld metals, was the more tortuous grain boundaries in the FM-82<sup>12-13</sup>. The SEM analysis revealed that this increase in grain boundary tortuosity is directly related with the large eutectic carbides present in the FM-82 weld metal. The GB tortuosity in FM-82 is due to the GB pinning imposed by these large (Nb, Ti)C carbides. The large (TiN) nitrides have a similar pinning effect in the FM-52 weld metal. However, the effect of these nitrides on the overall GB migration was not relevant due to the low fraction of precipitates and their uneven distribution. On the other hand, it may be argued that the large amount of medium and small intergranular and intragranular precipitates would have a more effective GB pinning effect, which is true. What must be considered in the case of the precipitates is if they were in the microstructure at the time most of the GB migration was occurring. The large (Nb, Ti)C precipitates in the FM-82 are eutectic carbides, which formed at the end of the solidification. The refractory TiN, which are thought to be transferred from the filler wire, were always present in the solid material, and therefore both, the eutectic carbides and the refractory nitrides, will have an important effect on GB migration during cooling following solidification. However, the small amount and uneven distribution of large nitrides in the FM-52 samples reduced their effect on GB migration and consequently GB tortuosity. On the other hand, the small and medium size precipitates observed on the grain boundaries, were precipitated in the solid state, after extensive GB migration had already occurred.

The SEM and TEM observations strongly suggest that DDC is creep-like in nature, as initially proposed by Rhines and Wray<sup>9</sup>. A variety of data now shows that DDC is a GB sliding, creep phenomena, which occurs at very high temperatures and under high stresses. Regarding the effect of the precipitates in this GB sliding creep process, they have a double effect. If precipitates are present in the microstructure at the time most of the GB migration would occur, their effect on increasing GB tortuosity will consequently reduce GB sliding and finally, increase the DDC resistance of the weld metal. The precipitates may reduce the creep deformation, but at the same time they act as sites for cavity nucleation<sup>14-15</sup>. However, the effect of intergranular particles on GB sliding and cavity nucleation strongly depends on the particle's nature, size, and distribution, of which the interrelationship among these factors is not totally understood. More research is

being conducted to clarify the actual effect of the medium and small intergranular precipitates in the DDC phenomena of Ni-base filler metals.

## Conclusions

The characterization work conducted to date suggests that DDC is a GB sliding, creep-like phenomenon, which is manifested at intermediate temperatures where the GB sliding process is activated. This sliding extends until reaching very high temperatures where dynamic recrystallization (DRX) occurs. Local strain mapping by EBSD analysis has shown that DRX prevents the excessive deformation accumulation around the grain boundaries and triple points, interrupting the creep fracture initiation.

The proposed DDC mechanism explains the difference in DDC susceptibility between FM-52 and FM-82, as revealed by the STF test, and clearly relates this behavior with some of the different features in the microstructures. However, other aspects that have not yet been fully analyzed, as the actual effect of the medium and small intergranular precipitates and GB segregation, may be playing important roles in the failure mechanism. This activity is part of the continuing effort to clearly describe the mechanism.

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