Improving the Ductility Dip Cracking Resistance of Ni-base Alloys

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Abstract. *Many austenitic alloys undergo a severe ductility drop at intermediate temperatures, which may result on the solid state phenomenon known as ductility dip cracking (DDC). This intermediate temperature intergranular cracking has been recognized as a grain boundary sliding creep-like phenomenon. Several factors have been identified to control the DDC susceptibility of these austenitic alloys, including grain size, grain boundary orientation to the applied force, impurity segregation to the grain boundaries, grain boundary tortuosity, and intergranular precipitation. This work studies the effect of Nb and Ti additions to Ni-base filler metals 52(ERNiCr-3) and 82(ERNiCrFe-7) on the DDC susceptibility. Strain-to-fracture test were performed on Ni-base filler metals 52 (0.25%Mn-8.9%Fe-29.1%Cr-0.5%Ti-0.7%Al-Ni) and 82 (2.75%Mn-0.7%Fe-20.1%Cr-2.6%Nb-0.5%Ti-Ni) with different amounts of Nb and Ti additions. Optical and electron microscopy were used to characterize the microstructures and fracture surfaces. Segregation of the added elements to the solidification grain and subgrain boundaries was verified. The Nb addition to FM-52 caused the precipitation of eutectic NbC carbides. The additions of Ti caused the formation of large Ti(C,N) and the additions of Ti and Nb caused the formation of (TiNb)(CN) eutectic-like precipitates in both filler metals. When large amounts of Nb and Ti were added to FM-52, colonies of acicular and blocky precipitates were observed. Solidification cracks formed when large amounts of Nb and/or Ti were added. On the other hand, small Nb and Ti additions caused an important reduction of DDC susceptibility. The Nb- and Ti-rich precipitates pinned the migrated GBs causing an increase in the GB tortuosity. For the temperatures and strains tested, Nb and Ti additions resulted in a general increase in the threshold strain required to initiate solid state cracking during the STF test.*
Keywords: *Ductility Dip Cracking, Nickel Base Alloys, Intergranular Precipitates, Nb, Ti*

1. Introduction

Some austenitic alloys undergo a severe ductility drop at temperatures between 0.5 and 0.7 of their melting temperature. When these materials are subjected to processing within this critical temperature range, a solid state intergranular cracking phenomenon known as ductility dip cracking (DDC), may become a serious and difficult production problem to overcome. For example, if alloys susceptible to this phenomenon are welded under high restraint conditions, the available ductility of the material may be exceeded by the welding-induced strain, causing welding induced DDC. This intermediate temperature cracking has been recognized as a grain boundary sliding creep-like phenomenon[1, 2, 3]. Several factors have been identified to control the DDC susceptibility of these austenitic alloys, including:

- Grain size;
- Weld metal chemical composition including impurity and interstitial elements;
- Segregation to the grain boundaries;
- Precipitation behavior;
- Grain boundary migration and pinning;
- Grain boundary orientation (macroscopic) relative to the applied strain;
- Grain boundary tourtousity;
- Dynamic recrystallization.

Among all the previously listed factors there is one that has been identified to have a strong influence on the DDC susceptibility of filler metals 52 and 82 welding deposits. This factor is the grain boundary tortuosity. Previous work by the authors, based on strain-to-fracture test [4], produced results which have shown that grain boundary tortuosity may be one of the most effective ways to reduce the intermediate temperature cracking of these specific Ni-base weld metals [5, 6]. This work presents recent studies of the effect of Nb and Ti additions on the DDC susceptibility of Ni-base filler metals 52 and 82.

The Filler metal 52 and 82 susceptibility to DDC has been throughly studied by the authors due to the importance of these alloys for the chemical, oil, and nuclear industries. Figure 1 presents the strain-to-fracture results for these alloys. The ductility of the alloys during the strain-to-fracture test is measured by the number of cracks formed during the deformation imposed at the test temperature.

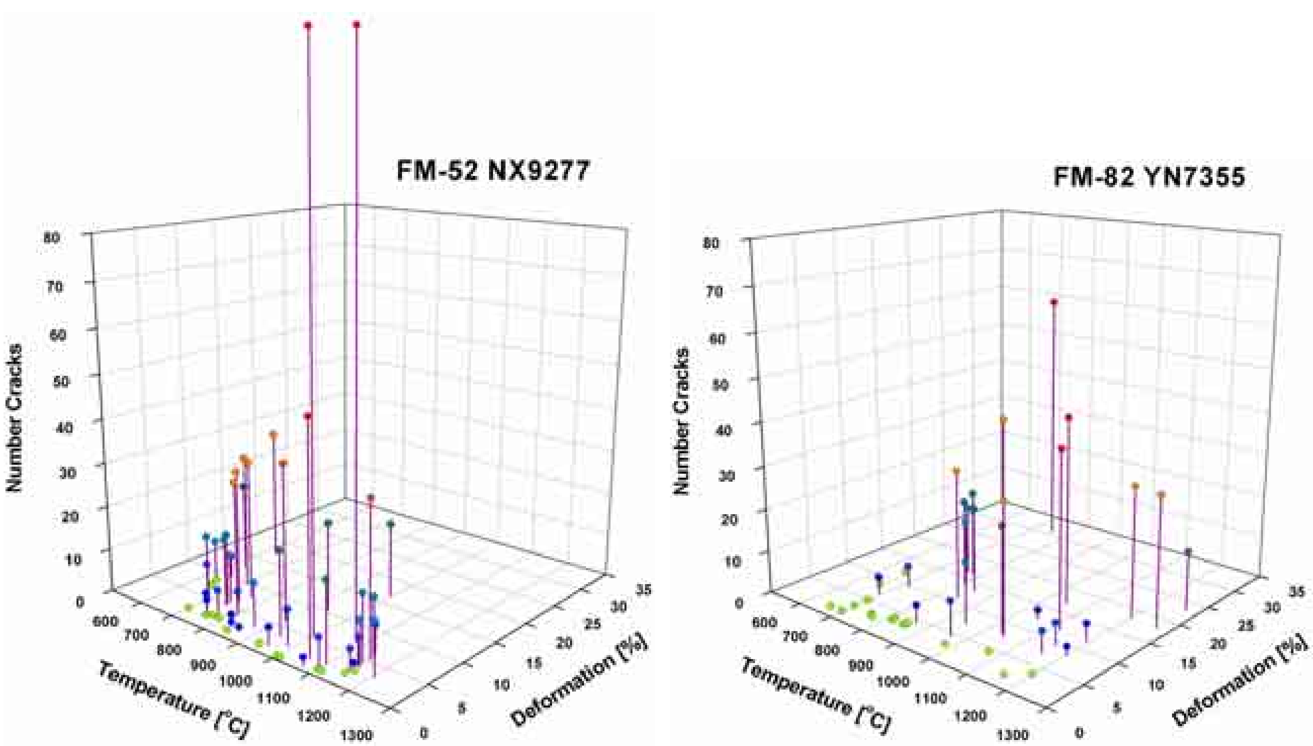

Figure 1. Strain-to-fracture test results for filler metal 52 and 82 weld deposits [5].

2. Experimental Procedure

Strain-to-fracture samples of Ni-base filler metals 52 and 82 have been prepared according to the recommendations of Nissley and Collins [4-5]. The chemical composition of the commercial filler metals is presented in Table 1. Different amounts of Nb and Ti were added to the weld metal to evaluate their influence on the high temperature ductility of these alloys. The optimized strain-to-fracture (STF) test was performed on the Gleeble® 3800 thermo-mechanical simulator at 950 °C, which is approximately the middle of the ductility trough for these alloys, as can be observed in Fig. 1. Additionally, the tested samples were metallographically prepared and analyzed on the light microscope and SEM. The fracture surfaces were also analyzed on the SEM.

Table 1. Chemical composition (wt%) of the filler metals (balance Ni)

Alloy	C	Mn	Fe	Cr	Nb	Ti	Al	Si	Cu	Mo	Co	S	P	Pb
FM-52 Heat NX9277	0.026	0.25	8.88	29.1	0.02	0.50	0.71	0.17	0.01	0.05	-	0.004	0.004	0.0001
FM-82 Heat YN7355	0.040	2.75	0.70	20.1	2.6	0.47	-	0.07	0.07	-	0.04	0.002	0.01	0.002

3. Results and Discussion

Segregation of the added elements to the solidification grain and subgrain boundaries was verified on the SEM using backscattered electron images and EDS measurements, as shown in Fig. 2. The Nb and Ti additions control the amount, morphology, and size of the carbides and carbo-nitrides precipitated in these alloys. The Nb addition to FM-52 caused the precipitation of eutectic NbC carbides. The additions of Ti caused the formation of large Ti(C,N),as shown in Fig. 3, and the additions of Ti and Nb caused the formation of (TiNb)(CN) eutectic-like precipitates in both filler metals. When large amounts of Nb (~2.0%wt) and Ti (~2.5%wt) were added to FM-52, colonies of acicular and blocky precipitates were observed, as presented in Fig. 4.

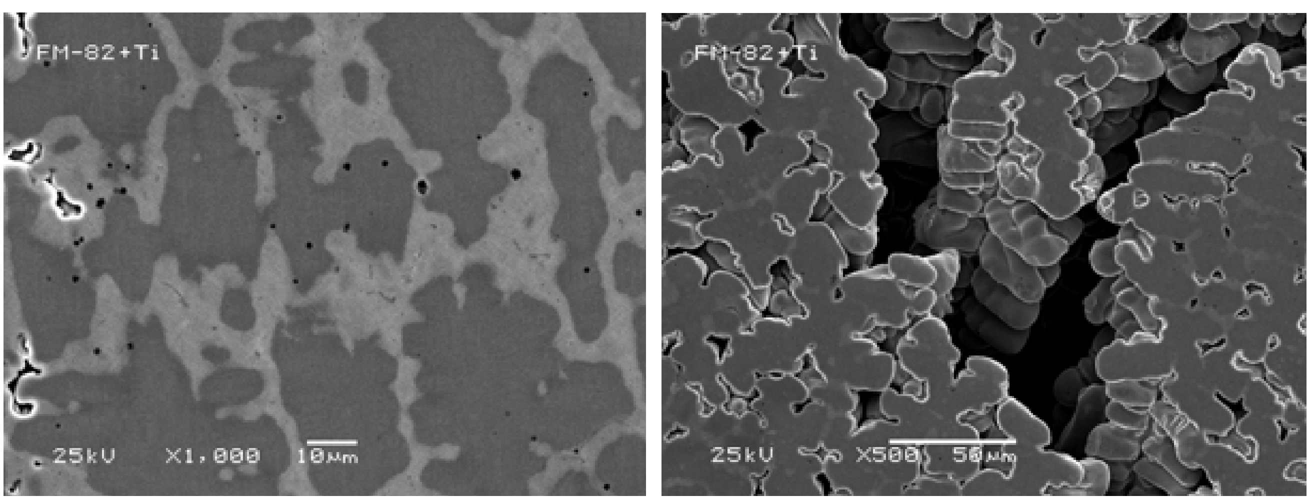

Figure 2. Solidification segregated microstructure and cracks in FM-82 weld metal with 2.5%wt Ti addition.

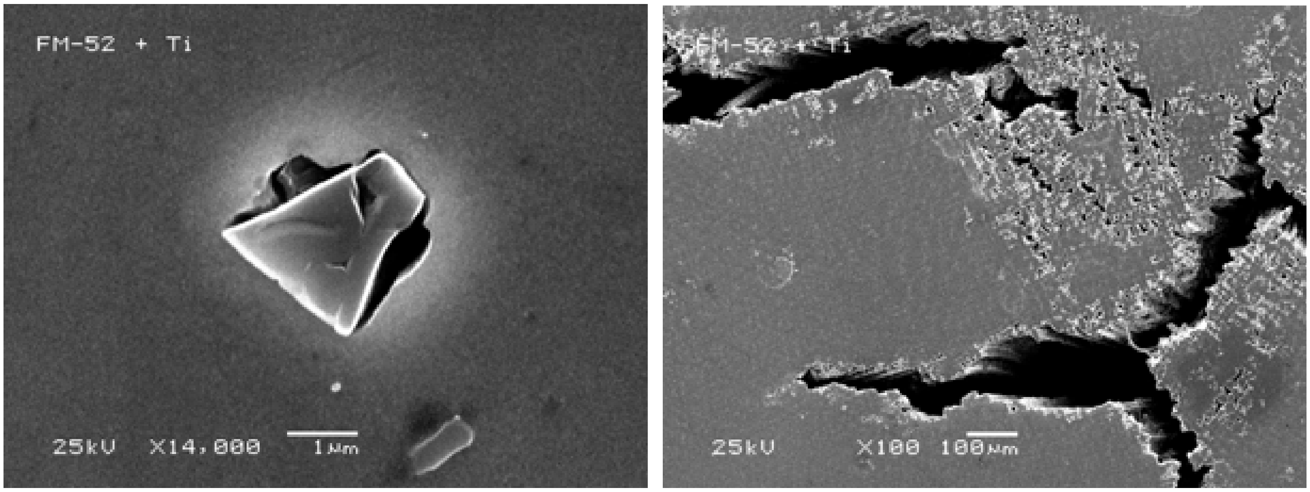

Figure 3. Ti(C,N) precipitated in FM-52 with large (3.5%) Ti additions.

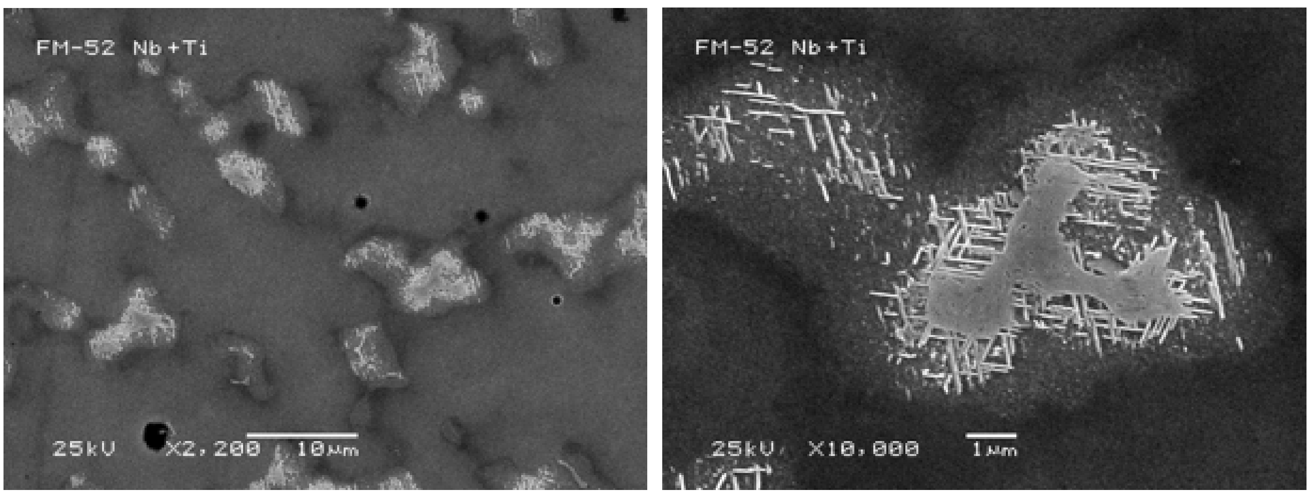

Figure 4. Blocky and acicular precipitate colonies within the interdendritic regions of FM-52 weld metal with Ti and Nb additions.

Liquid-related grain boundary cracks formed during the preparation of STF samples when large amounts of Nb (~ 2.0 wt%) and/or Ti (~ 3.5 wt%) were added to the weld metal, see Fig. 2, 3 and 5. Figure 2 shows a crack surface on the FM-82 STF sample with 2.5 wt% Ti addition. Figure 3 shows abundant cracking in the FM-52 STF sample with 3.5 wt% Ti addition. Figure 4 presents a crack surface in FM-52 weld metal with 2.0 wt% Nb addition. The previously mentioned cracks are clearly liquid related cracks, as evidenced by their dendritic morphology. The reheating at 950 °C to which these samples were submitted is not considered enough to cause liquation cracking. In addition, the size and morphology of these cracks does not suggest liquation cracking. Thus, these cracks are most likely solidification cracks formed along the solidification grain boundaries. The cracks were not noticed on the STF samples before the thermo-mechanical tests were performed. Thus, it is assumed that the preexisting cracks were opened during the STF test.

A second batch of samples was prepared with lower Nb and Ti additions. Solidification cracking did not occur among these samples. The STF test results showed some cracking susceptibility reduction.

Fig. 6 presents the comparison of the STF results between the weld metals produced with commercial FM-52 and 82 and their modified versions with low Ti additions respectively. The microstructural evaluation of the Ti-modified weld metals revealed two differentiated large Ti-rich precipitates. The first type are Ti(CN) and the other type are interdendritic (unidentified) Ti-rich precipitates as presented in Fig. 7. The precipitation caused in both filler metals by the Ti addition is far from what has been previously suggested as ideal [6]. These Ti-rich precipitates are considered too large to effectively pin grain boundaries during the primary cooling following solidification. However, the ductility improvement is evident.

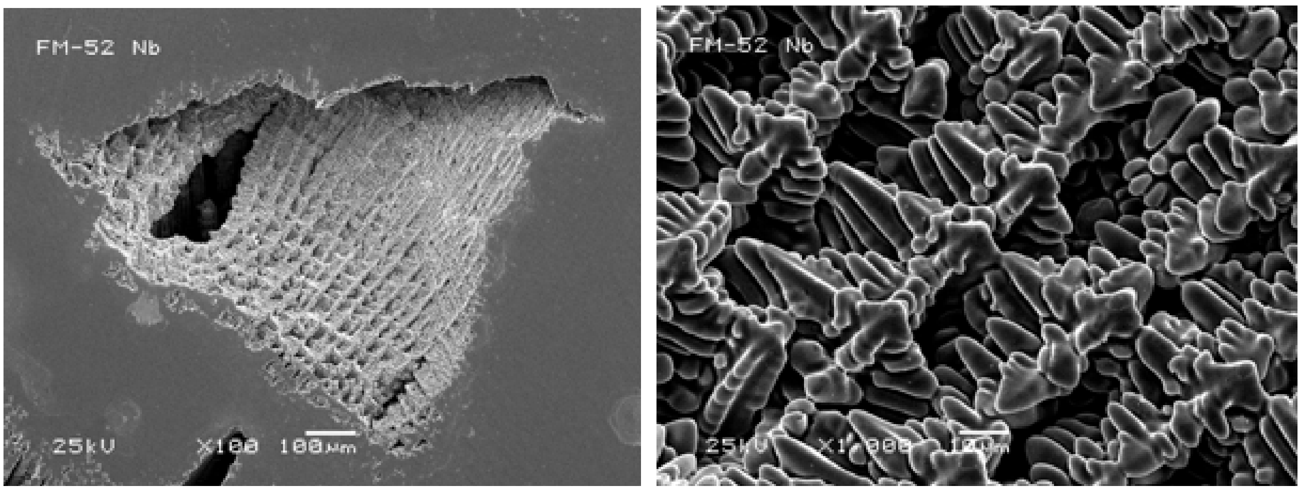

Figure 5. Solidification crack surface in FM52 with 2.0%wt Nb addition.

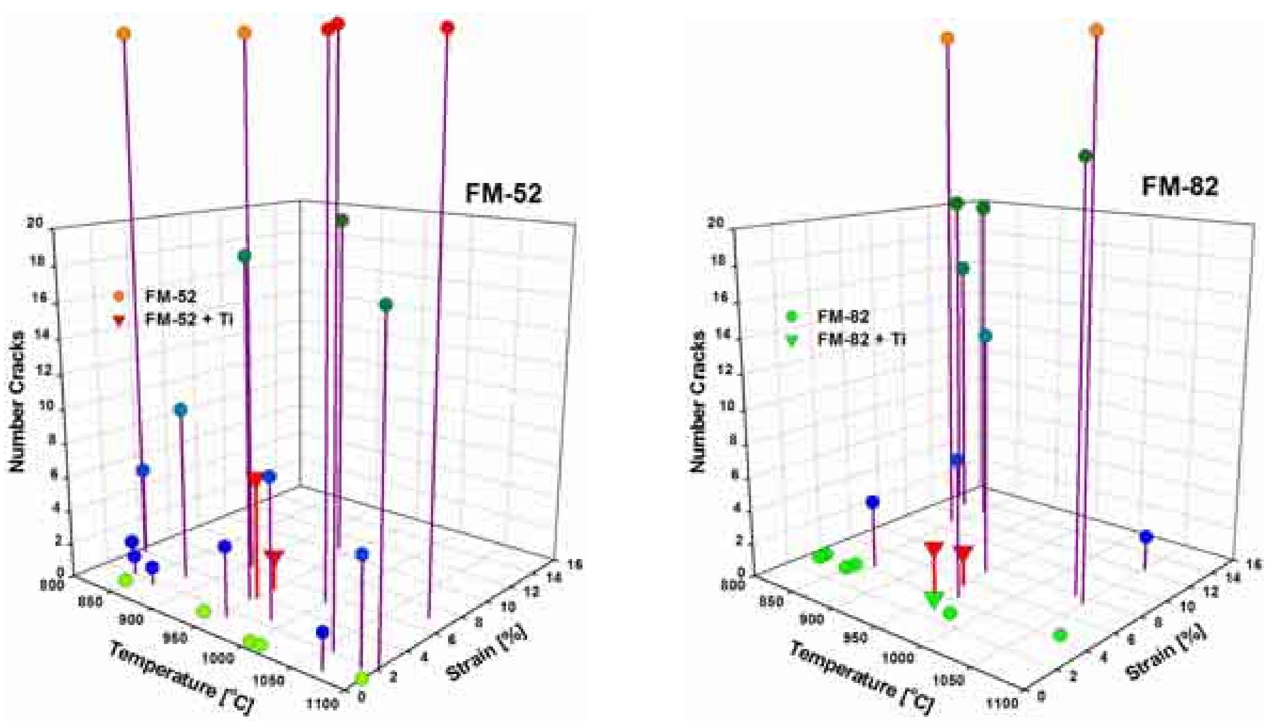

Figure 6. Comparison of STF test results on FM-52 and 82 without (circles) and with (triangles) low Ti additions.

On the other hand, the low Nb additions to an originally Nb-free FM-52, caused the precipitation of medium sized (NbTi)(CN), as presented in Fig. 8. The Nb addition to this alloy, made its microstructure very similar to the FM-82 microstructure. The medium sized Nb-rich precipitates effectively pinned the grain boundaries in this weld metal during the primary cooling following solidification, as normally happens with FM-82. The grain boundary pinning during the primary cooling originated very tortuous grain boundaries. This microstructure combining non-acicular medium size precipitates and tortuous grain boundaries resulted in improved material ductility at intermediate temperatures, as revealed by the STF test results presented in Fig. 9.

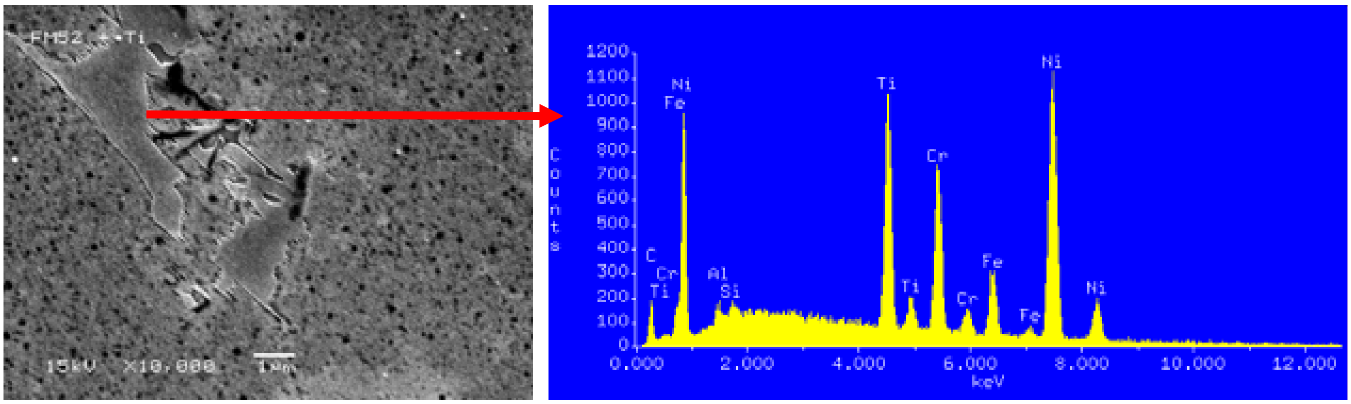

Figure 7. Ti-rich precipitates in FM-52 with low Ti additions.

When low levels of Nb+Ti were added to FM-52 weld metal a further improvement on intermediate temperature ductility was achived, as presented in Fig. 9. This improvement on ductility appears to be related to the observed precipitate refinement caused by the synergic interaction between the Nb and Ti additions. Figure 10 presents the microstructure of FM-52 weld metal modified with low Nb+Ti additions. This figure also presents a detailed micrograph of one Ti-Nb-rich precipitate and its EDS spectrum. Smaller, more numerous, and homogeneusly distributed precipitates on the interdendritic regions of this low Nb+Ti modified FM-52 weld metal resulted in the formation of very tourtous grain boundaries. In adition, the size and round-like morphology of these precipitates are considered important factors contributing to the ductility improvement.

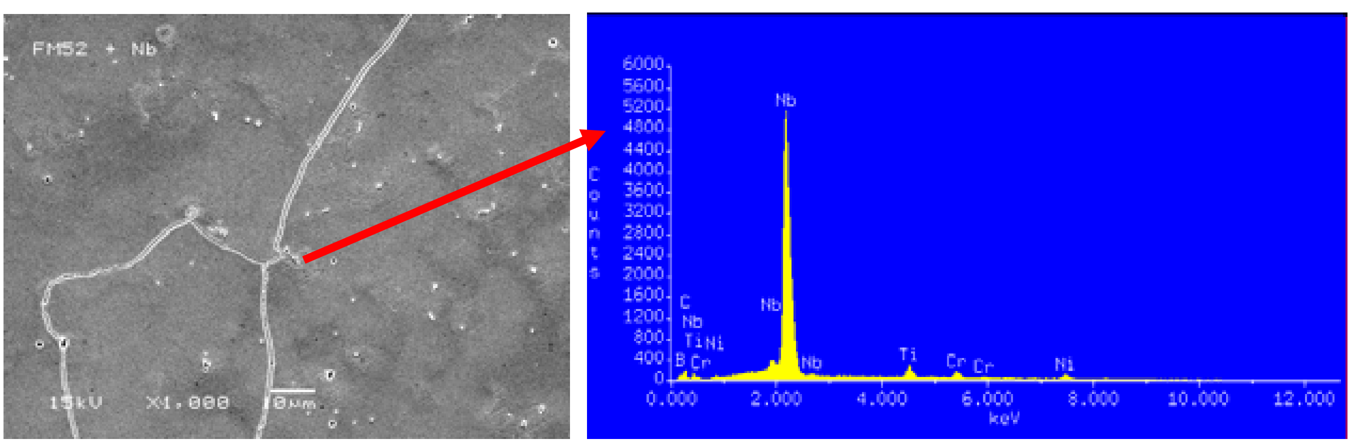

Figure 8. Microstructure of STF sample of FM-52 with low Nb aditions. EDS spectrum of Nb-rich precipitate.

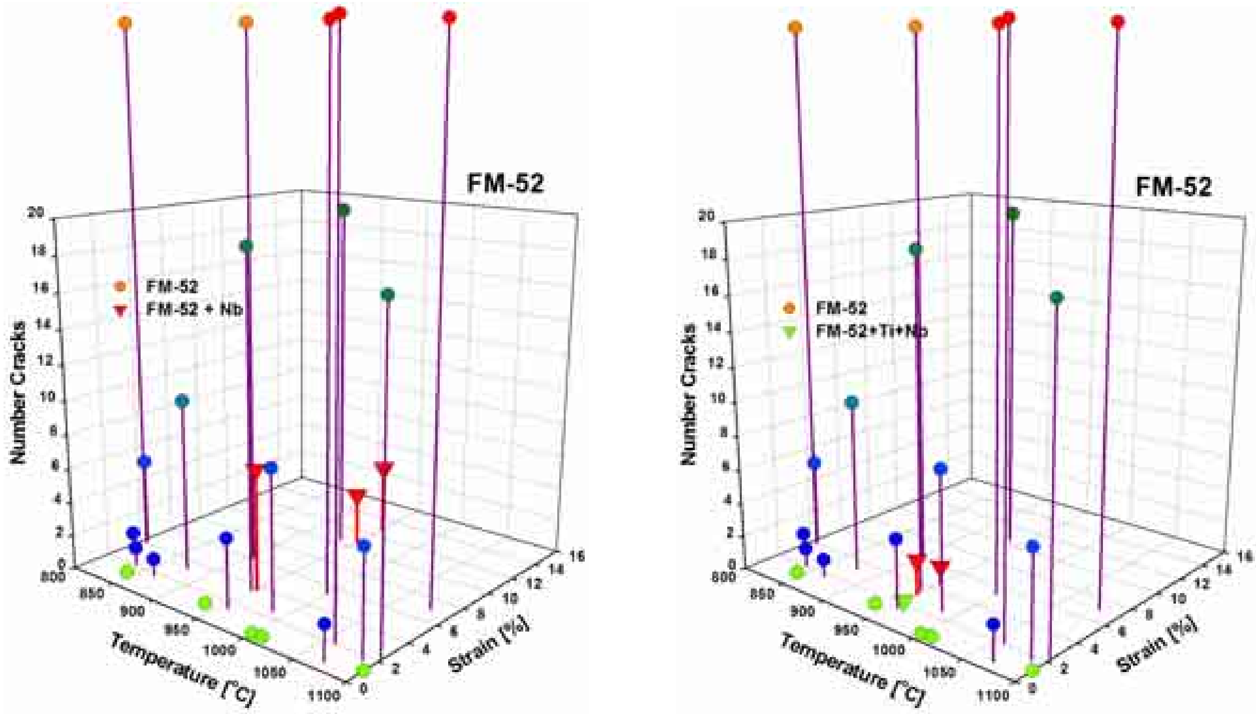

Figure 9. Comparison of STF test results on FM-52 with (triangles) low Nb and Nb+Ti additions.

In all cases, the Nb- and/or Ti-rich precipitates pinned the migrated grain boundaries causing an increase in the grain boundary tortuosity. For the temperatures and strains tested, Nb and Ti additions resulted in a general increase in the threshold strain required to initiate solid state cracking (ductility-dip cracking) during the STF test. The Ti addition appears to have a slightly superior effect than Nb. The difference in behavior between Nb and Ti modified alloys is believed, at this time, to be related to the type of precipitate that is formed. Since the Ti-rich carbo-nitrides form at higher temperature they may produce a more efficient pinning effect. However, the morphology of the Ti-rich precipitates does not appear to be the better. The Nb+Ti additions appear to have a synergic effect as previously discussed. More STF testing over a wider temperature and strain range is necessary to fully understand the effect of these additions on the DDC resistance. Additionally, further characterization on the STF samples will be conducted to obtain a more detailed characterization of the intergranular precipitates and their interaction with the grain boundary sliding phenomenon.

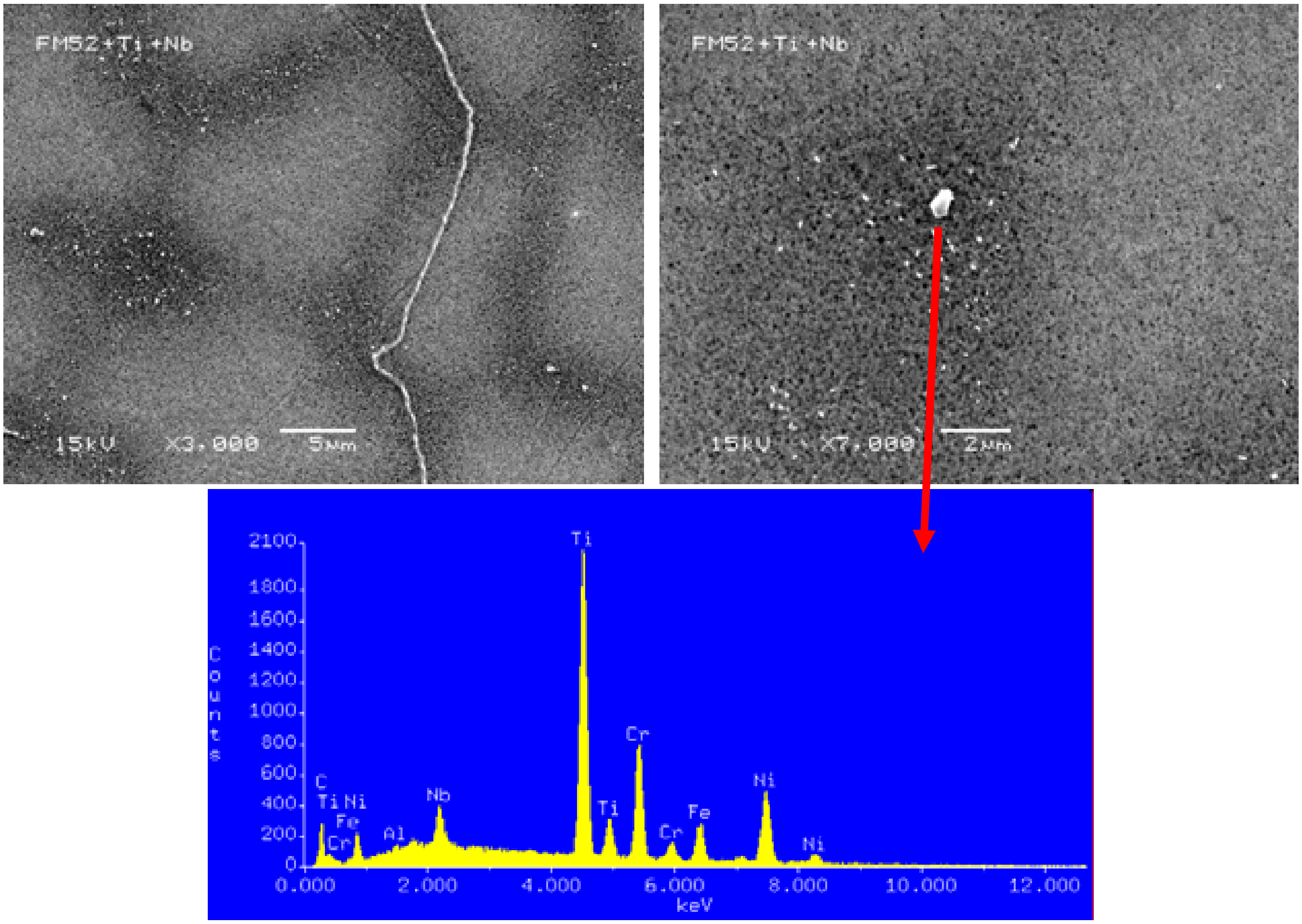

Figure 10. Microstructure of FM-52 STF sample with low Ti and Nb aditions. EDS spectrum of Ti-Nb-rich precipitate.

4. Conclusions

The additions of strong carbide and nitride forming elements such as Nb and Ti have an important effect on the filler metal 52 and 82 weld metal microstructure. Excessive additions of such elements to the weld metal caused solidification cracking. On the other hand, small additions have a largely beneficial effect on these alloys' DDC resistance. The improvement in DDC resistance is directly related to the GB morphology changes caused by the precipitates formed during solidification or at elevated temperature. These results provide new insight into the development of modified 52 and 82 DDC resistant filler metals.

5. Acknowledgments

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6. References

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