

## CHAPTER 13 - EXPERIMENT 11

### CHARACTERIZATION OF THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ADH BRAZE JOINTS IN FSX-414 COBALT-BASE SUPERALLOY NOZZLE SEGMENTS USING NOVEL BRAZE ALLOYS

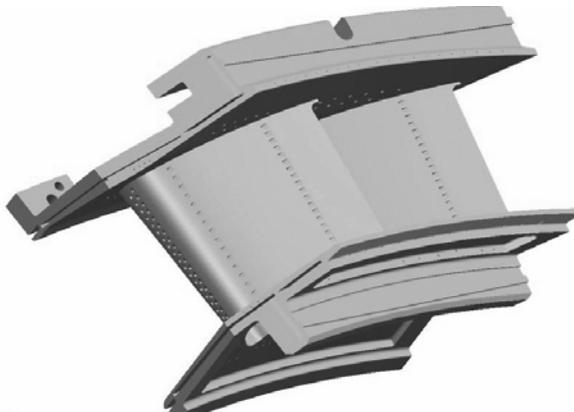
#### 13.1) Introduction

The preceding chapters focused on the use of binary and ternary Ni-Zr and Ni-Hf braze alloys for the repair of turbine components cast from Ni-base superalloys, such as Inconel 738 and MarM247. The promising mechanical properties of the novel braze joints in Ni-base superalloy components prompted an investigation into the use of novel braze alloys for repairing Co-base superalloy nozzle segments.

GE Energy Services, an OEM, currently produce a range of Industrial Gas Turbine (IGT) engines, including models such as the Frame7FA, Frame 7FA+ and Frame 7FA+e engines. All these engines contain first stage nozzle segments cast from FSX-414 cobalt-base superalloy. The chemical composition of FSX-414 is given in **Table 55**. This alloy is a solid solution strengthened alloy, in contrast to the  $\gamma'$  strengthened Ni-based superalloys (such as Inconel 738 and MarM247) considered in earlier chapters. A schematic illustration of a Frame7FA+e first stage nozzle segment with two airfoils is shown in **Figure 168**.

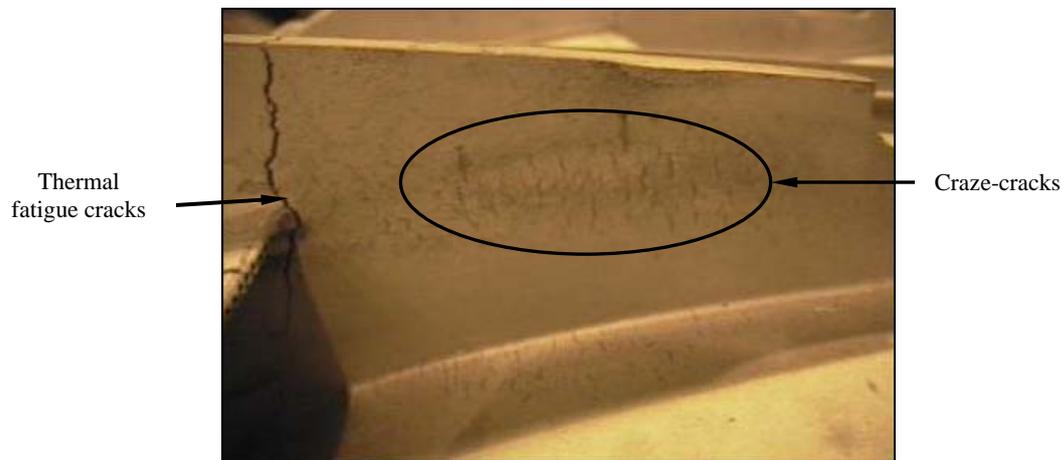
**Table 55** – Nominal chemical compositions of the alloys considered in this investigation (wt.%).

Alloy	Co	Cr	Ni	W	Fe	Ta	Zr	Si	Mn	Ti	Al	Ce	C	B
FSX-414	Bal	29.0	10.0	7.5	1.0	-	-	-	-	-	-	-	0.25	0.01
Nozzalloy	Bal	29.0	20.0	-	-	7.0	0.01	-	-	-	-	0.06	0.05	-
X-40	Bal	25.5	10.0	7.5	-	-	-	0.7	0.7	-	-	-	0.5	-
D15	10.3	15.3	Bal	-	-	3.5	-	-	-	-	3.0	-	-	2.5
MarM509	Bal	23.5	10.0	7.0	-	3.5	0.5	-	-	0.2	-	-	0.6	-
MarM509B	Bal.	23.5	10.0	7.0	-	3.5	0.5	0.2	-	0.2	-	-	0.6	2.5
MarM918	Bal	20.0	20.0	-	-	7.5	0.1	-	-	-	-	-	0.05	-



**Figure 168** - Schematic illustration of the first stage nozzle segment of a Frame7FA+e IGT engine.

Nozzle segments in IGT engines tend to degrade during engine service, with degradation modes ranging from craze cracking, oxidation, corrosion and creep, to foreign object damage. Examples of characteristic cracks in a first stage nozzle segment are shown in **Figure 169**. The cracks in this nozzle segment were originally repaired using manual arc welding with processes such as GTAW (gas tungsten arc welding) or PAW (plasma arc welding), and a Co-base filler metal known as Nozzalloy. The chemical composition of Nozzalloy is given in **Table 55**.



**Figure 169** - An example of craze-cracks and large individual thermal fatigue cracks on the outer sidewall of a first stage nozzle segment.

Although as a solid solution strengthened alloy, FSX-414 is readily weldable, the extent of cracking displayed in **Figure 169** resulted in several hours of weld time. Extensive welding caused distortion of the nozzle segments to the extent that key dimensions were outside allowable tolerance limits. This resulted in the nozzle segments not fitting properly on return to the engine. Since brazing in a vacuum furnace involves uniform heating and cooling, resulting in minimal distortion, a vacuum braze repair was developed. The patented wide gap braze process known as ADH (activated diffusion healing), described in §1.5, was used. The ADH repair involved mixing equal parts (by weight) X-40 cobalt-base superalloy powder and a  $\gamma'$  strengthened Ni-base braze alloy known as D15 (containing 2.5% B as melt point depressant). The nominal compositions of X-40 and D15 are given in **Table 55**. The X-40/D15 mixture resulted in a hybrid braze mix with poor mechanical properties, and the joint cracked rapidly in service. As a result of the poor mechanical integrity of the braze joints, customers refused to have their nozzle segments ADH braze repaired, and insisted that the nozzle segments be welded.

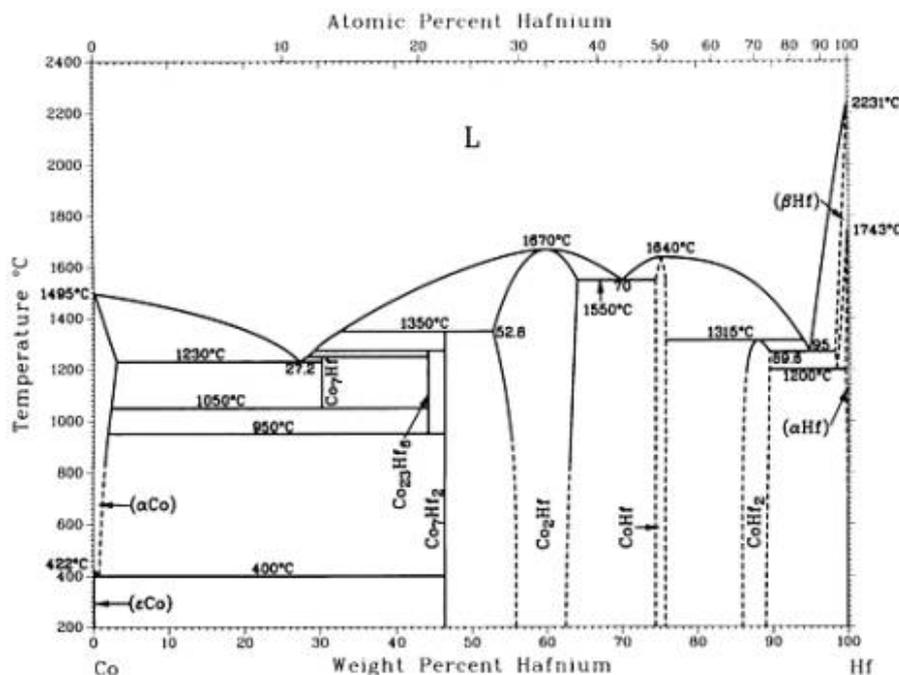
The poor service performance of the X-40/D15 ADH braze repair prompted further investigation with the following objectives:

- To examine an alternative ADH braze mixture consisting of MarM509 Co-base superalloy powder and MarM509B braze alloy for crack repair of FSX-414 nozzle segments, with the aim of obtaining mechanical integrity equivalent or superior to the properties of Nozzalloy welds.
- To develop and test a novel Co-base braze alloy containing Hf instead of B as melt point depressant for the crack repair of FSX-414 nozzle segments.

Three properties are considered critical with respect to the quality of the braze joints: elevated temperature tensile properties, creep rupture properties and low cycle fatigue life. If a newly developed braze repair procedure ensures mechanical properties equivalent to those of a weld repair, the improved braze procedure would most likely be preferred by repair customers due to the lower risk of distortion.

In view of the stated requirements for an acceptable braze procedure, the X-40/D15 ADH braze mixture was changed to a MarM509/MarM509B braze. The chemical compositions of MarM509 and MarM509B are shown in **Table 55**. MarM509 is a Co-base superalloy with an excellent combination of mechanical properties, including high tensile strength, fatigue strength and creep rupture strength. MarM509B is a Co-base braze alloy with a nominal chemical composition virtually identical to that of MarM509, with 2.5 wt.% B added as melt point depressant. Certification documents for the MarM509B braze alloy assign a solidus temperature of 1121°C and a liquidus temperature of 1160°C to the alloy. Si is present as a trace element in MarM509B and is not added intentionally to lower the melting point.

The promising results described in earlier chapters for the binary and ternary eutectic Ni-Hf braze alloys encouraged the development of a novel Co-base braze alloy containing Hf as melt point depressant. The binary Co-Hf phase diagram [2], shown in **Figure 170**, illustrates that a eutectic point exists at a composition of 27.2% Hf and 72.8% Co (by weight), and a temperature of 1230°C. This investigation therefore included an evaluation of a novel ADH braze joint consisting of a mixture of MarM509 powder and the binary eutectic Co-Hf braze alloy.

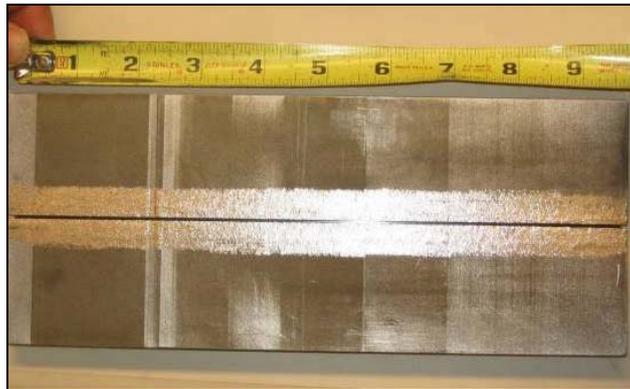


**Figure 170** – The binary Co-Hf phase diagram [2].

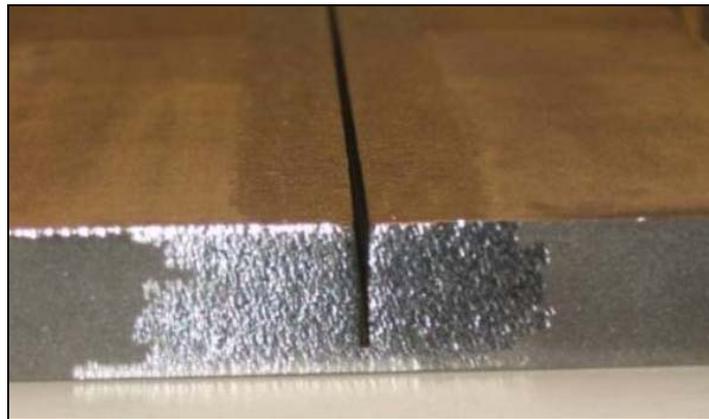
### 13.2) Experimental procedure

Plates of FSX-414 with dimensions 248 mm long, 127 mm wide and 19 mm thick were investment cast for this experiment. A 1.5 mm wide groove was machined in the center of each plate, as illustrated in **Figure 171**. This gap width was selected on the basis of an investigation into the crack widths observed in 96 nozzle segments (four engine sets, with 24

nozzle segments in each engine set). The data revealed a mean crack width of 0.64 mm and a maximum crack width of 1.5 mm. The approval authority required that the new ADH process be capable of repairing up to the maximum crack width, i.e. 1.5 mm. The gap was only machined partially, leaving 0.5 mm of parent metal intact at the base of the groove (as shown in **Figure 172**). This was done to maintain the 1.5 mm gap width during brazing without the need for special fixturing. Since the mechanical test samples were machined to a gauge diameter of 6.4 mm, the 0.5 mm parent metal retained at the base of the groove was machined away after brazing and therefore did not affect the test results in any way.



**Figure 171** - The FSX-414 test plate with a 1.5 mm wide groove machined in the centre.



**Figure 172** - Enlarged view of the machined groove in the braze test plate.

Two sets of braze samples were produced. The first set of samples was produced by filling the groove with a mixture of MarM509 powder and MarM509B braze powder (with compositions shown in **Table 55**). The powders were mixed in a ratio of 60% MarM509 and 40% MarM509B (by weight). The second set of samples was produced by filling the groove with a mixture of MarM509 powder and eutectic Co-Hf powder (containing 27.2% Hf) in a ratio of 60% MarM509 powder and 40% braze alloy. The plates were then processed in a vacuum furnace using the following braze cycle:

- 1) Evacuate the vacuum furnace to a pressure of  $5 \times 10^{-4}$  torr or lower.
- 2) Ramp up to a temperature of  $450^{\circ}\text{C} \pm 14^{\circ}\text{C}$  at a rate between  $8^{\circ}\text{C}/\text{minute}$  and  $14^{\circ}\text{C}/\text{minute}$ .
- 3) Hold at  $450^{\circ}\text{C}$  for a minimum of 40 minutes to allow the binder to burn off. Proceed to the next step once the vacuum level had returned to  $1 \times 10^{-3}$  torr or lower.

- 4) Ramp up to a temperature of  $1093^{\circ}\text{C}\pm 14^{\circ}\text{C}$  at a rate between  $8^{\circ}\text{C}/\text{minute}$  and  $14^{\circ}\text{C}/\text{minute}$ .
- 5) Hold at  $1093^{\circ}\text{C}$  for a minimum of 40 minutes to allow the samples to stabilize at this temperature.
- 6) Ramp up to the melting temperature of  $1245^{\circ}\text{C}\pm 14^{\circ}\text{C}$  at a rate between  $8^{\circ}\text{C}/\text{minute}$  and  $14^{\circ}\text{C}/\text{minute}$ .
- 7) Hold at  $1245^{\circ}\text{C}$  for a minimum of 30 minutes.
- 8) Furnace cool under vacuum to  $1150^{\circ}\text{C}\pm 14^{\circ}\text{C}$ .
- 9) Hold at  $1150^{\circ}\text{C}$  for a minimum of 240 minutes.
- 10) Ramp up to a temperature of  $1177^{\circ}\text{C}\pm 14^{\circ}\text{C}$  at a rate between  $8^{\circ}\text{C}/\text{minute}$  and  $14^{\circ}\text{C}/\text{minute}$ .
- 11) Hold at  $1177^{\circ}\text{C}$  for a minimum of 240 minutes.
- 12) Ramp up to a temperature of  $1205^{\circ}\text{C}\pm 14^{\circ}\text{C}$  at a rate between  $8^{\circ}\text{C}/\text{minute}$  and  $14^{\circ}\text{C}/\text{minute}$ .
- 13) Hold at  $1205^{\circ}\text{C}$  for a minimum of 240 minutes.
- 14) Gas quench with argon or helium gas to a safe temperature (about  $90^{\circ}\text{C}$ ) before unloading the test plates.

A melting temperature of  $1245^{\circ}\text{C}$  was selected as it is above the liquidus temperatures of both the MarM509B ADH braze alloy and the novel Co-Hf braze alloy. The diffusion temperatures ( $1150^{\circ}\text{C}$ ,  $1177^{\circ}\text{C}$  and  $1205^{\circ}\text{C}$ ) were selected to be below the liquidus temperatures of both braze alloys, but high enough to facilitate rapid diffusion of the melt point depressants, i.e. B or Hf, into the surrounding superalloy powder particles.

Photographs of the MarM509/Co-Hf and the MarM509/MarM509B ADH joints are shown in **Figures 173(a) and (b)**. The appearance of the joints suggests that the MarM509/MarM509B ADH braze alloy was more fluid than the MarM509/Co-Hf braze at the melting temperature. This is attributed to the amount of superheating above the liquidus temperature of the braze alloy, with the MarM509/MarM509B ADH braze being processed at a temperature  $68^{\circ}\text{C}$  higher than its liquidus temperature, whereas the MarM509/Co-Hf ADH braze was processed at a temperature only  $16^{\circ}\text{C}$  higher than its eutectic temperature.



**Figure 173** - Photographs of: (a) the MarM509/Co-Hf ADH test plate in the as-brazed condition; and (b) the MarM509/MarM509B ADH test plate in the as-brazed condition.

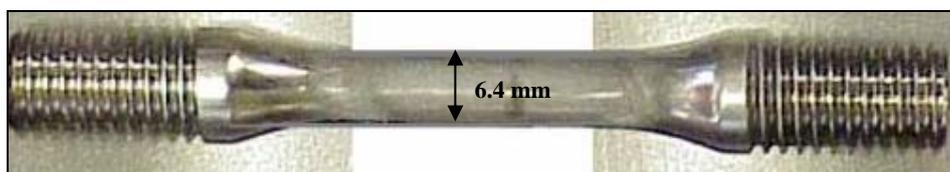
In order to compare the mechanical properties of the novel ADH braze joints with those of the welds currently preferred by repair customers, MarM509 plates were fusion welded using a

double V-weld preparation in the downhand position. The GTAW (gas tungsten arc welding) process was used with the following weld parameters:

- Welding current: 60 to 80 A
- Arc voltage: 10 to 15 V
- Welding speed: Manual
- Tungsten electrode: 2% Thoriated tungsten (classification: EWTh-2), 1.6 mm or 2.4 mm diameter
- Polarity: DCEN (direct current electrode negative)
- Filler wire diameter: 0.9 mm to 1.6 mm
- Shielding gas: 95% Ar + 5% H<sub>2</sub> (by volume)
- Preheat temperature: 10°C minimum
- Interpass temperature: 150°C maximum
- Welding technique: Stringer bead
- Gas cup size: 9.5 mm to 19 mm
- Filler metal: Nozzalloy or MarM918 (nominal chemical compositions are shown in **Table 55**).

Tensile tests, creep rupture tests and low cycle fatigue (LCF) tests were performed at various temperatures. An example of a mechanical test sample machined from the test plates is shown in **Figure 174**. The elevated temperature tensile tests were performed at 870°C. Creep rupture tests were performed at 760°C, 815°C, 870°C, 980°C, 1040°C and 1090°C at various applied stress levels. The LCF tests were performed at 870°C at various strain levels. Since the success of this investigation hinged mainly on the LCF properties of the joints, the Approval Repair Authority requested that the following LCF curves be generated:

- a) FSX-414 base metal welded with MarM918 filler metal. This weld filler metal is used to salvage virgin castings with casting defects, such as hot tears or macroporosity. The chemical composition of MarM918 is given in **Table 55**.
- b) FSX-414 welded with Nozzalloy filler metal. This weld filler metal is used to repair nozzle segments degraded during service.
- c) FSX-414 brazed with the new MarM509/MarM509B wide gap braze mixture.
- d) FSX-414 brazed with the new MarM509/Co-Hf ADH braze mixture.



**Figure 174** - Mechanical test sample machined from the braze test plates. The 1.5 mm wide braze joint is located in the centre of the gauge length.

### 13.3) Results and discussion

#### 13.3.1 Mechanical test results:

The results of tensile tests performed at 870°C are shown in **Table 56** for the FSX-414 base metal, FSX-414 welded with Nozzalloy, FSX-414 ADH brazed with MarM509/MarM509B, and FSX-414 ADH brazed with the novel Co-Hf braze alloy.

**Table 56** - Tensile properties at 870°C of FSX-414 base metal, FSX-414 welded with Nozzalloy, FSX-414 brazed with MarM509/MarM509B, and FSX-414 brazed with MarM509/Co-Hf. The test samples were produced with a joint gap of 1.5 mm to simulate a worst-case crack repair scenario.

Specimen identification	Failure location	Tensile strength, MPa	Yield stress, MPa	% Elongation	% Reduction in area
BLT-1T (FSX-414 base metal)	Base metal	291	168	32	32
BLT-2T (FSX-414 base metal)	Base metal	304	177	31	31
BLT-3T (FSX-414 base metal)	Base metal	296	167	44	44
BLT-4T (FSX-414 base metal)	Base metal	283	164	36	36
<b>Average for the FSX-414 base metal</b>	-	<b>295</b>	<b>169</b>	<b>35.8</b>	<b>35.8</b>
NOZ-1 (FSX-414 welded with Nozzalloy)	HAZ*	278	175	44	53
NOZ-7 (FSX-414 welded with Nozzalloy)	Base metal	274	179	46	45
NOZ-10 (FSX-414 welded with Nozzalloy)	Base metal	275	177	42	52
NOZ-16 (FSX-414 welded with Nozzalloy)	Base metal	275	178	38	43
<b>Average for the FSX-414/Nozzalloy weld</b>		<b>275</b>	<b>177</b>	<b>42.5</b>	<b>48.3</b>
509-1 (FSX-414 brazed with MarM509/MarM509B)	Base metal	277	190	48	46
509-7 (FSX-414 brazed with MarM509/MarM509B)	Base metal	282	189	28	44
509-10 (FSX-414 brazed with MarM509/MarM509B)	Base metal	268	178	30	38
509-16 (FSX-414 brazed with MarM509/MarM509B)	Base metal	284	179	29	48
<b>Average for the MarM509/MarM509B ADH joint</b>		<b>277</b>	<b>184</b>	<b>33.8</b>	<b>44.0</b>
ENH-1 (FSX-414 brazed with MarM509/Co-Hf)	Base metal	272	175	28	51
ENH-7 (FSX-414 brazed with MarM509/Co-Hf)	Base metal	268	175	50	59
ENH-10 (FSX-414 brazed with MarM509/Co-Hf)	Base metal	285	194	25	56
ENH-16 (FSX-414 brazed with MarM509/Co-Hf)	ADH joint	275	190	4.3	4.3
<b>Average for the MarM509/Co-Hf ADH joint</b>		<b>275</b>	<b>183</b>	<b>34.3</b>	<b>55.3</b>

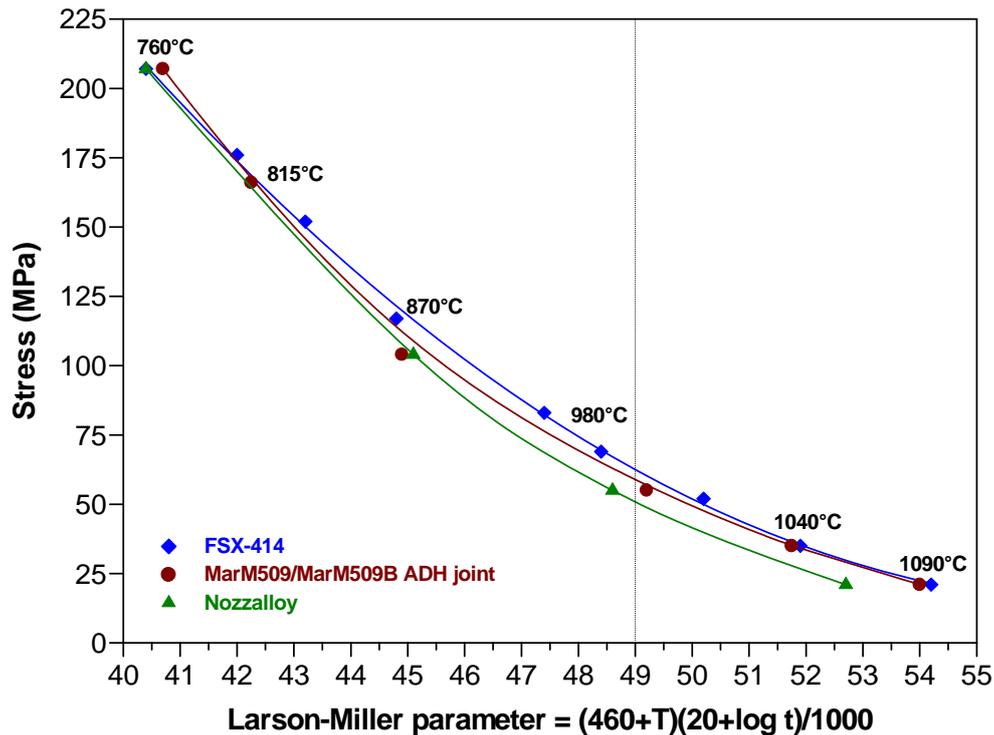
\* HAZ: Heat-affected zone.

It is evident from the tensile test results shown in **Table 56** that the FSX-414 samples fusion welded with Nozzalloy filler metal performed very well, with three of the four samples tested failing in the parent metal and the fourth failing in the heat-affected zone. The tensile strength, yield strength, elongation and reduction in area values were equivalent or even superior to those of the base metal.

FSX-414 brazed with the MarM509/MarM509B ADH material yielded similar results, with all the tensile samples failing in the base metal. The tensile test results shown in **Table 56** therefore represent the properties of the base metal, rather than those of the braze joint. It can, however, be concluded that the tensile properties of the MarM509/MarM509B braze joints were at least equivalent to those of the base metal.

The FSX-414 samples brazed with the MarM509/Co-Hf braze alloy yielded promising results, with three of the four samples failing in the base metal. It can be concluded that the tensile properties of these joints were at least equivalent to those of the base metal. The fourth sample failed in the braze joint, but displayed tensile and yield strength values equivalent to those of the base metal (as shown in **Table 56**). The elongation and reduction in area values of the fourth sample were, however, very low when compared with those of the base metal (4.3% elongation and reduction in area, compared with  $\pm 35\%$ ). Examination of the fractured sample revealed porosity on the fracture surface. It is assumed that the low ductility values measured during this tensile test were related to the presence of porosity in the braze joint.

A Larson-Miller plot comparing the creep rupture properties of the FSX-414 base metal, FSX-414 welded with Nozzalloy filler metal, and FSX-414 ADH brazed with MarM509/MarM509B, is shown in **Figure 175**. The Larson-Miller plot indicates that the MarM509/MarM509B braze samples outperformed the Nozzalloy welds. For example, at a Larson-Miller number of 49 (the dotted line in **Figure 175**), the MarM509/MarM509B samples exhibited 90% of the base metal's creep rupture properties, whereas the Nozzalloy samples displayed 75% of the base metal's properties. At higher test temperatures the creep rupture properties of the MarM509/MarM509B braze joints approached those of the base metal. On average the MarM509/MarM509B samples demonstrated 90% of the base metal's creep rupture properties, whereas the Nozzalloy samples displayed 72%.



**Figure 175** – Larson-Miller plot of FSX-414 base metal, the MarM509/MarM509B ADH braze joints, and the Nozzalloy weld.

The results of the low cycle fatigue (LCF) tests are shown in **Table 57**, and displayed graphically in **Figure 176**.

Although MarM918 filler metal is approved for salvaging virgin castings, all except one of the FSX-414 samples welded with MarM918 failed in the weld metal or heat-affected zone. The MarM918 welds displayed the poorest fatigue life of all the samples evaluated. The Nozzalloy welds performed marginally better, with four of the twelve samples failing in the weld or heat-affected zone. The remaining eight Nozzalloy samples failed in the base metal.

The MarM509/MarM509B braze joints displayed consistently higher LCF properties than the welded joints, with four of the twelve samples failing at the interface between the braze joint and the base metal. The remaining eight samples failed in the base metal.

**Table 57** – Low cycle fatigue (LCF) data for FSX-414 base metal, FSX-414 welded with MarM918, FSX 414 welded with Nozzalloy, FSX-414 ADH brazed with MarM509/MarM509B, and FSX-414 ADH brazed with MarM509/Co-Hf.

Sample	Strain	Cycles to initiation	Cycles to failure	Failure location
FSX-414 base metal	0.60	1298	1460	Base metal
	0.60	1598	2099	Base metal
	0.50	2978	3563	Base metal
	0.50	3206	3773	Base metal
	0.46	6177	6375	Base metal
	0.40	4422	5787	Base metal
	0.40	7126	8765	Base metal
	0.35	8584	9087	Base metal
	0.35	11085	12972	Base metal
	0.33	14290	17141	Base metal
	0.30	22736	24712	Base metal
0.30	49953	51066	Base metal	
FSX-414 welded with MarM918	0.60	814	1013	Weld
	0.60	1212	1398	Weld
	0.50	1669	2039	HAZ*
	0.50	2153	2955	HAZ*
	0.45	1301	2056	HAZ*
	0.40	4194	4539	Weld
	0.40	3876	5302	Weld
	0.40	4885	5312	Weld
	0.35	8308	8768	Weld
	0.35	12006	12310	HAZ*
	0.30	10058	12310	Weld
0.30	20999	26395	Base metal	
FSX-414 welded with Nozzalloy	0.60	1920	1957	Base metal
	0.60	1871	2649	Base metal
	0.50	3036	3618	HAZ*
	0.50	2655	3294	Base metal
	0.45	2009	2357	Base metal
	0.40	2316	2924	Weld
	0.40	4989	6583	HAZ*
	0.40	6895	7163	Base metal
	0.35	6569	7372	HAZ*
	0.35	9947	11526	Base metal
	0.30	18243	20239	Base metal
0.30	20206	22163	Base metal	
FSX-414 ADH brazed with MarM509/MarM509B	0.69	989	1007	Interface**
	0.60	1156	1238	Interface**
	0.60	876	1360	Base metal
	0.50	1854	1912	Interface**
	0.50	2641	3010	Base metal
	0.45	3893	4939	Base metal
	0.44	4890	5289	Base metal
	0.40	3735	5189	Base metal
	0.40	5173	5556	Base metal
	0.35	8036	9188	Interface**
	0.35	11911	14986	Base metal
0.30	26421	30221	Base metal	

Table 57 – continued.

Sample	Strain	Cycles to initiation	Cycles to failure	Failure location
FSX-414 ADH brazed with MarM509/Co-Hf	0.70	1195	1588	Base metal
	0.60	1627	1970	Base metal
	0.60	2711	2927	Base metal
	0.50	2134	2248	Base metal
	0.50	3589	3914	Base metal
	0.45	3501	3898	Base metal
	0.45	3092	3906	Base metal
	0.40	4975	5771	Base metal
	0.40	5455	6849	Base metal
	0.35	16584	17726	Base metal
	0.35	23185	25365	Base metal
	0.30	40911	41754	Base metal

\* HAZ: Heat-affected zone  
\*\* Interface: Joint line between the base metal and the ADH braze

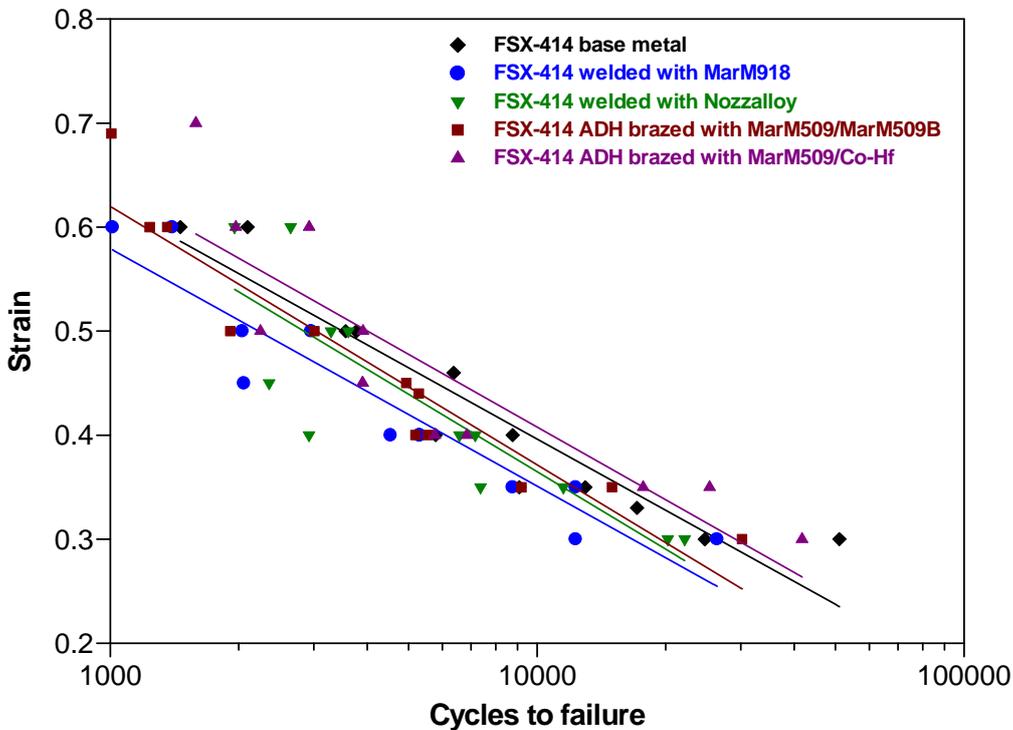


Figure 176 – Graphical representation of the LCF data determined for FSX-414 base metal, FSX-414 welded with MarM918 or Nozzalloy filler metal, FSX-414 ADH brazed with MarM509/MarM509B, and FSX-414 ADH brazed with MarM509/Co-Hf.

Figure 176 demonstrates that the MarM509/Co-Hf braze joint outperformed all the other repairs during LCF testing. It is also important to note that all the MarM509/Co-Hf samples failed in the base metal, and that the ADH braze joint displayed LCF properties superior to those even of the FSX-414 base metal. No publication in open literature has ever reported consistent base metal failure for braze joints. This resulted in consensus in both the aircraft and industrial gas turbine repair industries that braze joints are inherently brittle, and fail rapidly under conditions of applied strain. Joints produced using braze alloys with B as melt

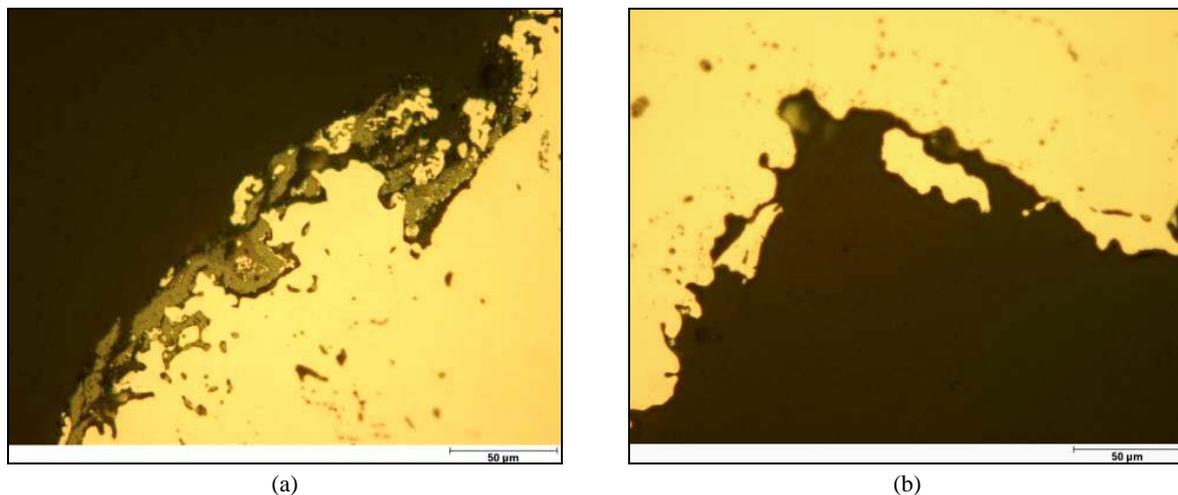
point depressant have been shown to fail consistently in the braze joint under LCF test conditions.

Statistical analysis of the LCF results revealed that FSX-414 welded with MarM918 suffered a 38% reduction in LCF life, compared to the base metal. FSX-414 welded with Nozzalloy displayed a 27% reduction in LCF life (for the four samples that failed in the weld metal or heat-affected zone), whereas the four FSX-414 samples brazed with MarM509/MarM509B that failed at the joint interface suffered a 10% reduction in LCF life. All the FSX-414 samples brazed with MarM509/Co-Hf displayed LCF properties superior to those of the base metal.

### 13.3.2 Metallurgical examination:

Nozzle segments (especially first stage nozzle segments) endure the highest temperatures in a gas turbine engine and are exposed to fluctuating thermal strains which may result in thermal fatigue cracking. During engine service these thermal fatigue cracks can manifest as individual cracks or clusters of cracks, with the latter usually referred to as craze-cracks (shown earlier in **Figure 169**).

Thermal fatigue cracks develop surface oxide layers during service which must be removed prior to ADH brazing. These oxide layers are usually comprised of Cr-oxides, Ni-oxides or a combination of these compounds. The hydrogen cleaning process effectively reduces these oxides within cracks in Co-base superalloys. Simply stated, the hydrogen cleaning process pulses H<sub>2</sub> into a furnace at elevated temperature (around 1200°C) for a time of between 8 and 12 hours. The surface oxides are reduced at elevated temperature in this hydrogen-containing environment. **Figures 177(a) and (b)** compare the sidewalls of a crack before and after hydrogen cleaning, and illustrate the effectiveness of the cleaning process in removing surface oxide layers.



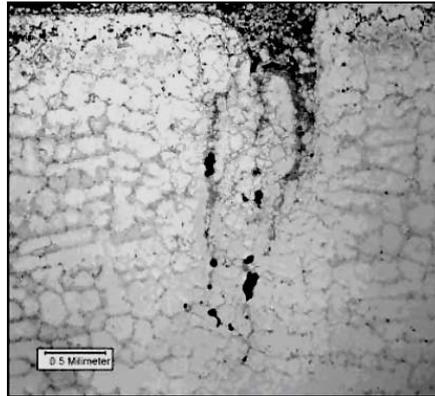
**Figure 177** – (a) Chromium oxides on the crack surface; and (b) oxide reduction on the sidewalls of the crack after H<sub>2</sub> cleaning.

**Figure 178** displays a macrograph of craze-cracks successfully repaired using the MarM509/MarM509B braze alloy. This photograph illustrates that more than eleven small cracks had been filled with braze alloy. **Figure 179** shows an individual crack, also filled

with the MarM509/MarM509B braze. Some porosity is evident, but it amounts to less than 10% (by volume), which is the acceptance limit.

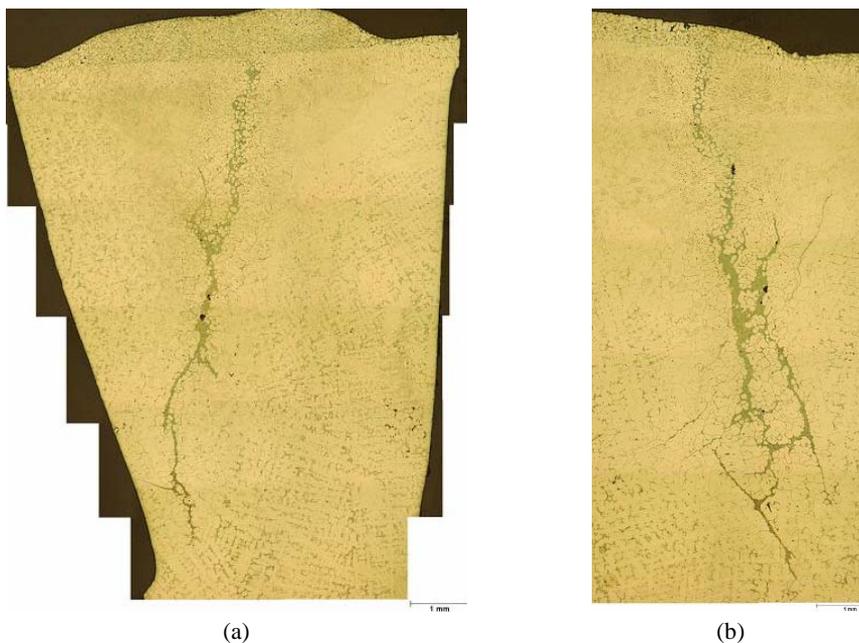


**Figure 178** - Craze-cracks filled with the MarM509/MarM509B braze alloy.

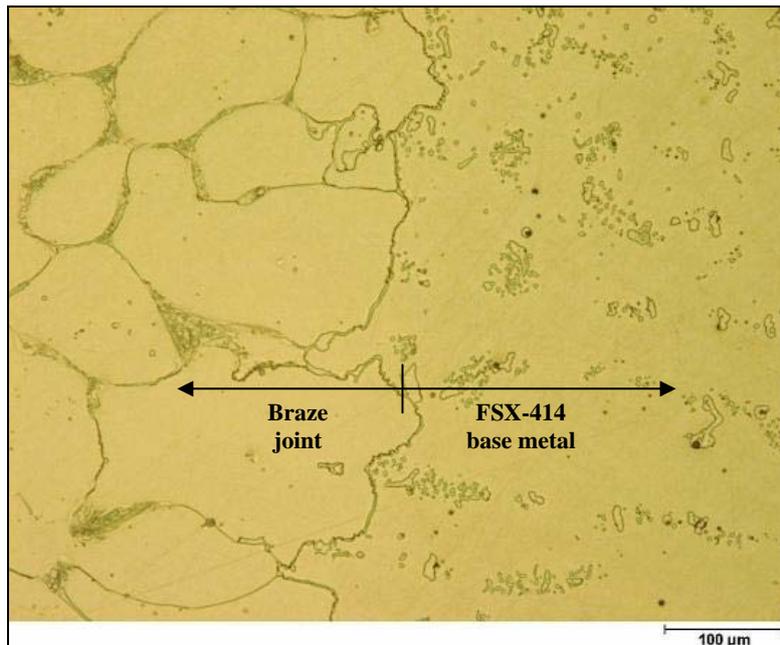


**Figure 179** - Individual crack filled with the MarM509/MarM509B braze alloy.

**Figures 180(a) and (b)** demonstrate the ability of the MarM509/MarM509B braze alloy to repair deep cracks which had propagated from the concave to the convex surface of an airfoil during service. The structure is undiffused, i.e. the repair process had only progressed to the melting stage (1245°C for 30 minutes). As shown in **Figure 181**, good bonding of the braze filler metal to the sidewalls of the crack was obtained.

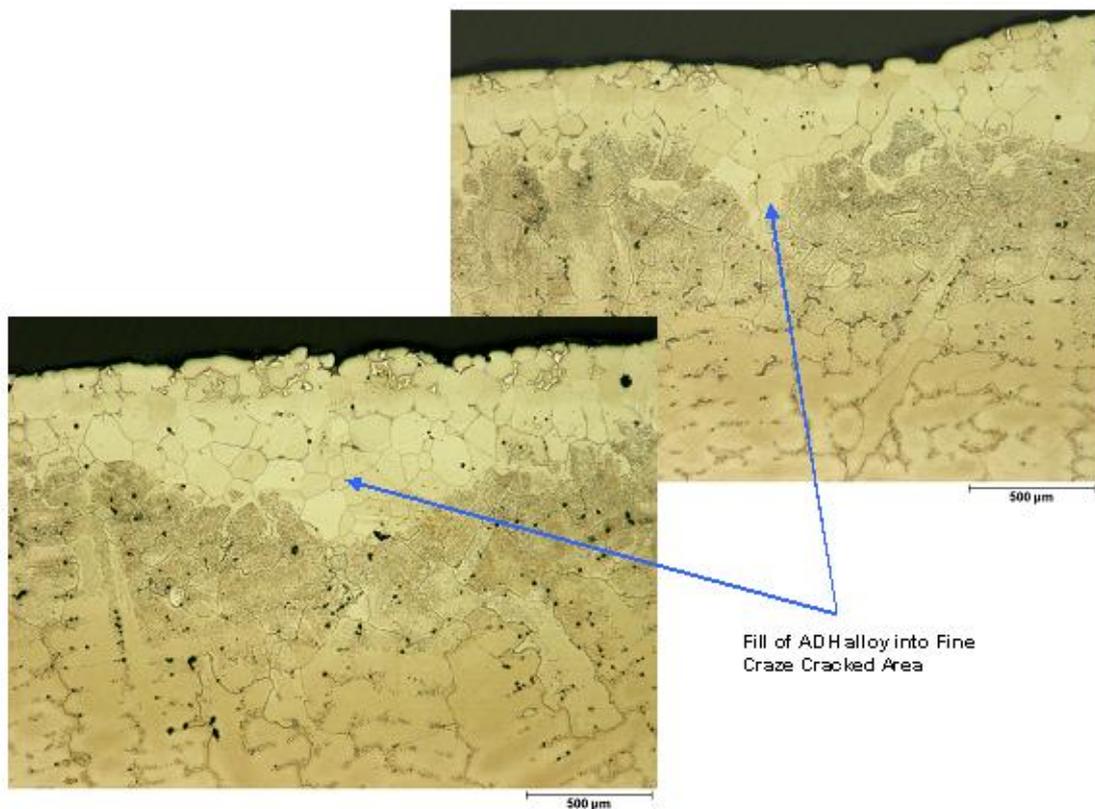


**Figure 180** – (a) Braze flow in a 7.5 mm deep crack; and (b) braze flow in an 11 mm deep crack.

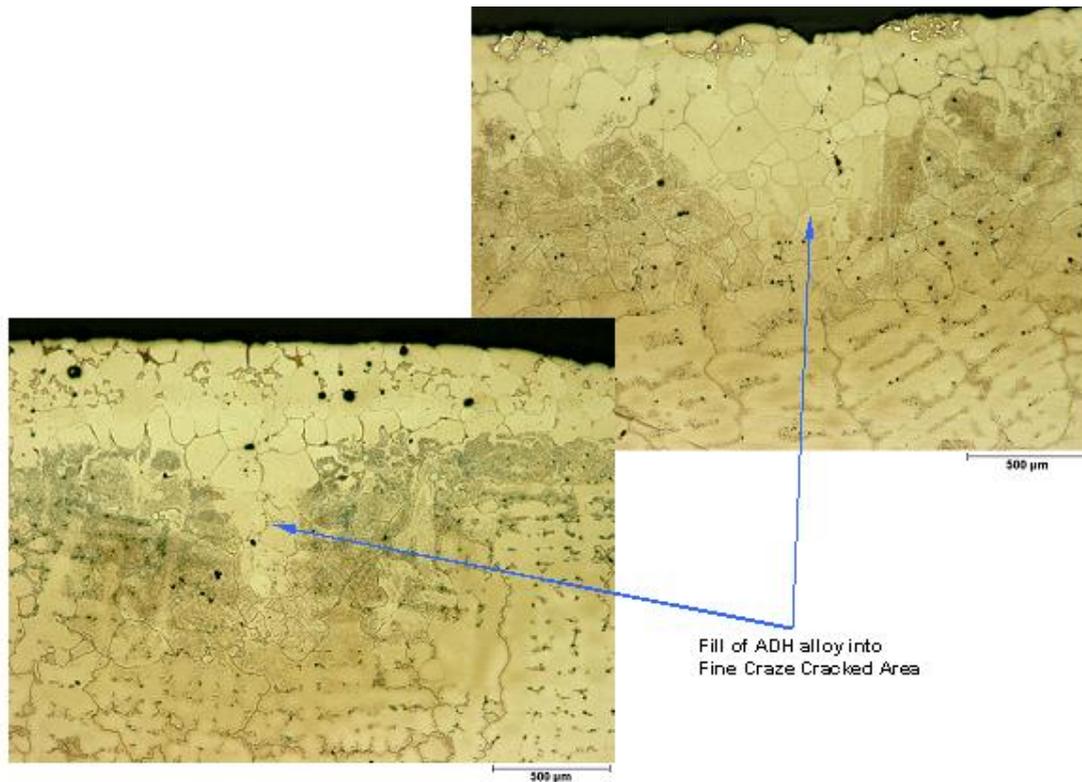


**Figure 181** – Photomicrograph of the interface between the braze crack repair and the base metal.

**Figures 182 and 183** display micrographs of braze joints used for craze-crack repair. These joints have undergone the full braze procedure, including the diffusion treatments. An equiaxed, homogenous structure, resembling that of the parent metal, is evident.



**Figure 182** - Microstructure of the fully diffused braze repair of craze-cracks on a nozzle segment.



**Figure 183** - Microstructure of the fully diffused braze repair of craze-cracks on a nozzle segment.

#### 13.4) Conclusions

- This experiment examined the use of alternative ADH braze mixtures consisting of MarM509 Co-base superalloy powder and MarM509B braze alloy, or MarM509 powder and a eutectic Co-Hf braze alloy, for crack repair in FSX-414 Co-base superalloy nozzle segments. The aim of the investigation was to obtain mechanical integrity equivalent or superior to the properties of the fusion welds currently preferred by repair customers.
- Both the eutectic Co-Hf braze alloy (consisting of 27.2% Hf and 72.8% Co by weight) and the MarM509B braze were effective in repairing cracks in FSX-414 Co-base superalloy nozzle segments. Since nozzle segments have been shown to contain cracks up to 1.5 mm in width, the Co-Hf and MarM509B braze alloys had to be mixed with MarM509 superalloy powder to facilitate a wide gap ADH braze repair.
- The hydrogen cleaning process was effective in reducing the oxide layers typically found in thermal fatigue cracks in FSX-414 nozzle segments. Although the MarM509/Co-Hf braze alloy proved to be more sluggish than the MarM509/MarM509B alloy during brazing, the braze alloys filled craze-cracks and individual cracks, and bonded effectively to the sidewalls of the cracks.
- ADH braze repair of FSX-414 using the novel MarM509/MarM509B and MarM509/Co-Hf braze alloys resulted in high temperature tensile properties equivalent to those of weld repairs in the same parent material (using Nozzalloy filler metal). The creep rupture and low cycle fatigue (LCF) properties of the braze joints were superior to those of welds performed using Nozzalloy or MarM918 filler metal.

- FSX-414 samples braze repaired using the novel MarM509/Co-Hf braze alloy consistently failed in the base metal under low cycle fatigue conditions, implying that the LCF properties of the braze joint were superior to those of the base metal. This suggests that failure is likely to occur preferentially in the base metal if cracks in FSX-414 nozzle segments are ADH braze repaired using this braze alloy. A braze alloy consisting of a mixture of 60% MarM509 powder (by weight) and 40% eutectic Co-Hf powder therefore shows potential for crack repair of nozzle segments in industry.
- Statistical analysis of the LCF results revealed that FSX-414 welded with MarM918 suffered a 38% reduction in LCF life compared to the base metal. FSX-414 welded with Nozzalloy displayed a 27% reduction in LCF life (for four samples that failed in the weld metal or heat-affected zone), whereas the four FSX-414 samples brazed with MarM509/MarM509B that failed at the joint interface suffered a 10% reduction in LCF life. All the FSX-414 samples brazed with MarM509/Co-Hf displayed LCF properties superior to those of the base metal.
- Microstructural examination of brazed and fully diffused MarM509/MarM509B crack repairs revealed equiaxed braze microstructures with few boride particles.

## **CHAPTER 14 – FINAL CONCLUSIONS**

This investigation was designed to:

- evaluate the use of two novel melt point depressants, namely Hf and Zr, in Ni- and Co-base braze filler metals, and
- optimize the liquid phase diffusion bonding process in conjunction with these novel braze filler metals with the aim of producing high strength joints with good ductility and mechanical properties at elevated temperature approaching those of the base metal.

This chapter describes the most important results obtained and the major conclusions drawn on completion of this project.

### **14.1 Liquid phase diffusion bonding of In738 Ni-base superalloy using binary eutectic Ni-Hf and Ni-Zr braze alloys**

During the course of this investigation, the merit of two novel melt point depressants, namely Hf and Zr, was evaluated and the liquid phase bonding process optimized for the repair of wide cracks in Ni-base superalloy turbine components. The following conclusions were drawn:

- Hf and Zr were found to act as effective melt point depressants in Ni-base braze alloys. Binary eutectic Ni-Hf and Ni-Zr braze alloys melt and flow satisfactorily when processed at a melting temperature of 1230°C. In the as-brazed condition, the braze alloys were found to consist of a nickel-rich  $\gamma$  phase and an intermetallic compound (provisionally identified as  $\text{Ni}_2\text{Hf}_7$  in the Ni-Hf braze alloy and  $\text{Ni}_5\text{Zr}$  in the Ni-Zr braze).
- In order to repair wide cracks in land-based turbine components, the binary Ni-Hf or Ni-Zr braze alloys need to be combined with a  $\gamma'$  strengthened Ni-base superalloy powder (such as MarM247) within the joint to form a liquid phase diffusion bond on brazing.
- A vacuum braze cycle consisting of melting at 1230°C, followed by an extended diffusion cycle of 24 hours at the same temperature, solution annealing at 1230°C, hot isostatic pressing at 1200°C and a full primary and secondary ageing cycle to precipitate  $\gamma'$  within the MarM247 particles, is recommended for obtaining optimal mechanical properties in the novel wide gap braze joints.
- Although the braze joints did not exhibit elevated temperature strength and creep rupture properties equivalent to those of the base metal, the novel wide gap braze joints consistently outperformed the commercially available B-containing braze alloys. The ductility of the novel braze alloys containing Hf or Zr as melt point depressant was significantly higher than the ductility of similar joints containing boron as melt point depressant.
- The Ni-Zr or Ni-Hf braze joints did not display LCF properties equivalent to those of the base metal, but the novel braze filler metals outperformed the commercially available B-containing brazes. Within the intermediate strain range where most cracks occur in nozzle segments (0.4% to 0.8% strain), the Ni-Zr and Ni-Hf braze joints displayed 70% and 74%, respectively, of the LCF properties of the base metal, compared to 11% for a B-containing braze alloy.

#### **14.2 Liquid phase diffusion bonding of In738 Ni-base superalloy using more complex Ni-Hf and Ni-Zr braze alloys alloyed with chromium**

In an attempt to further improve the performance of the novel braze filler metals, a series of more complex braze formulations containing chromium, in addition to various other deliberately added alloying elements, was produced. The microstructures of these formulations were examined in the as-cast, as-brazed or fully diffused condition, and electron microprobe analysis techniques were used to identify the phases observed in each alloy. The following conclusions were drawn:

- The addition of Cr to the novel Ni-Hf braze alloy shifted the eutectic composition to a lower Hf content.
- The boride particles which form in B-containing braze alloys were found to be considerably harder than the  $\text{Ni}_7\text{Hf}_2$  intermetallic phase that forms in the novel Ni-Cr-Hf braze alloys. This difference in hardness may account for the high ductility and good LCF properties of the Ni-Hf braze joints, compared to those of B-containing braze alloys.
- Electron microprobe analysis of braze joints in complex alloys containing Hf or Zr as melt point depressants revealed that the intermetallic phases which form in the joints are most likely  $\text{Ni}_5\text{Zr}$  in Zr-containing brazes, and the nonequilibrium  $\text{Ni}_7\text{Hf}_2$  phase in Hf-containing braze alloys. Dilution and contamination of the braze mixtures during production complicated identification of the intermetallic compounds.
- The high solidus and liquidus temperatures of many of the more complex alloys make them unsuitable for use in the repair of equiaxed Ni-base superalloy materials, such as In738 and MarM247. An alloy with a composition of Ni-8.4Cr-5.4W-1.6Ti-6.1Al-14.5Hf (wt.%), however, demonstrated great potential for the repair of equiaxed, directionally solidified and single crystal Ni-base superalloys.

#### **14.3 Liquid phase diffusion bonding of FSX-414 Co-base superalloy using two novel ADH braze mixtures**

The poor service performance of ADH braze joints currently used for refurbishing FSX-414 Co-base superalloy nozzle segments prompted an investigation into the use of alternative ADH braze mixtures in this application. The investigation examined ADH mixtures consisting of MarM509 Co-base superalloy powder and commercially available MarM509B braze alloy, and MarM509 powder and a eutectic Co-Hf braze alloy for FSX-414 crack repair, with the aim of obtaining mechanical integrity equivalent or superior to the properties of weld repairs. The following conclusions were drawn based on the results of the investigation:

- Both the eutectic Co-Hf braze alloy (consisting of 27.2% Hf and 72.8% Co by weight) and the MarM509B braze were effective in repairing cracks in FSX-414 Co-base superalloy nozzle segments. Since nozzle segments have been shown to contain cracks up to 1.5 mm in width, the Co-Hf and MarM509B braze alloys were mixed with MarM509 superalloy powder to facilitate a wide gap ADH braze repair.
- ADH braze repair of FSX-414 using the novel MarM509/MarM509B and MarM509/Co-Hf braze alloys resulted in high temperature tensile properties equivalent to those of weld repairs in the same parent material (using Nozzalloy filler metal). The creep rupture and low cycle fatigue (LCF) properties of the braze joints were superior to those of welds performed using Nozzalloy or MarM918 filler metal.

- Statistical analysis of the LCF results revealed that FSX-414 welded with MarM918 suffered a 38% reduction in LCF life compared to the base metal. FSX-414 welded with Nozzalloy displayed a 27% reduction in LCF life, whereas four FSX-414 samples brazed with MarM509/MarM509B failed at the joint interface and suffered a 10% reduction in LCF life.
- FSX-414 samples braze repaired using the novel MarM509/Co-Hf braze alloy consistently failed in the base metal under low cycle fatigue conditions, implying that the LCF properties of the braze joint were superior to those of the base metal. This suggests that failure is likely to occur preferentially in the base metal if cracks in FSX-414 nozzle segments are ADH braze repaired using this braze alloy. A braze alloy consisting of a mixture of 60% MarM509 powder (by weight) and 40% eutectic Co-Hf powder therefore has considerable potential for crack repair of nozzle segments in industry.

## **CHAPTER 15 – RECOMMENDATIONS FOR FUTURE WORK**

- The results of SEM-EDS and electron microprobe analyses of the braze joints suggested that the intermetallic compound observed in the binary and more complex Ni-Hf joints was the nonequilibrium  $\text{Ni}_7\text{Hf}_2$  phase, whereas the intermetallic phase observed in the Ni-Zr joints was most likely  $\text{Ni}_5\text{Zr}$ . In order to confirm the chemical composition and structure of the intermetallic phases observed in the braze joints, transmission electron microscopy work is recommended.
- Experiment 10, reported in Chapter 12, demonstrated that a complex Ni-base braze alloy with a composition of Ni-8.4Cr-5.4W-1.6Ti-6.1Al-14.5Hf (wt.%) (referred to as alloy PV9025) had solidus and liquidus temperatures below those of the simple eutectic Ni-Hf braze alloy evaluated in earlier experiments. This braze alloy therefore holds promise for the repair of equiaxed, directionally solidified and single crystal Ni-base superalloys. Since the microstructure of this complex Ni-base alloy was evaluated in the as-cast condition, it is recommended that the alloy be mixed with Ni-base superalloy powder and used to produce a wide gap liquid phase diffusion bond in a  $\gamma'$  strengthened Ni-base superalloy, such as In738. The properties of the braze samples should then be evaluated in the fully heat treated and aged condition. Such an evaluation should include examination of the joint microstructure using optical microscopy, scanning electron microscopy and electron microprobe techniques, and full mechanical testing (including elevated temperature tensile tests, creep rupture tests and low cycle fatigue tests).
- The results of Experiment 11, described in Chapter 13, revealed that ADH braze repair of wide cracks in FSX-414 cobalt-base superalloy nozzle segments using novel MarM509/MarM509B or MarM509/Co-Hf ADH braze mixtures resulted in excellent joint mechanical properties. The braze joints displayed tensile properties at elevated temperature equivalent to those of the base metal, and creep rupture properties superior to those of weld repairs in the same base metal. The low cycle fatigue properties of the MarM509/Co-Hf joints were shown to be at least equivalent to those of the FSX-414 base metal, with failure occurring in the base metal of all the joints evaluated. In order to further characterize these novel ADH braze alloys, it is recommended that the microstructures of MarM509/MarM509B and MarM509/Co-Hf joints be characterized using optical microscopy, scanning electron microscopy and electron microprobe analysis techniques.
- FSX-414, the Co-base superalloy currently used by GE Energy Services for casting nozzle segments, is recognized as having mechanical properties inferior to those of the Co-base superalloys used by a number of other original engine manufacturers (OEM's) for the same application. Siemens Westinghouse, an OEM, uses ECY 768 Co-base superalloy for nozzle segment casting, whereas Pratt & Whitney, Siemens and Alstom, also OEM's, use MarM509. Future work should evaluate the mechanical integrity of ADH braze joints produced using the novel MarM509/MarM509B and MarM509/Co-Hf ADH braze mixtures in these higher strength Co-base superalloys.