MICROSTRUCTURAL AND MECHANICAL PROPERTY CHARACTERIZATION OF AGED INCONEL ALLOY 625LCF.

by

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ABSTRACT

Industrial and aerospace applications for INCONEL® alloy 625LCF® sometimes require thermal exposure in the temperature range of 593°C to 650°C for extended periods of time. The alloy has the potential to precipitate both γ'' and $M_{23}C_6$ carbides in this temperature regime with concomitant changes in mechanical properties. This paper correlates observed microstructural changes as a function of time and temperature with selected properties for the alloy. Microstructural features as defined by microscopy, SEM and X-ray identification of extracted phases were utilized to characterize the microstructure after exposure at 593°C and 650°C for times to 7500 hrs (11 months). The same material was evaluated for its room and high temperature tensile properties. Observations from this study were compared to historical reports of microstructure and properties.

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Introduction

Since its introduction in 1989, INCONEL alloy 625LCF has grown in industrial acceptance. Applications are similar to those of conventional alloy 625 which is typically used in exhaust systems, thrust reversers, bleed air ducting, combustors, transition ducts and engine components.[1] Because of its optimized chemistry and improved fatigue resistance, alloy 625LCF is finding increased acceptance in these applications. Due to its finer grain size and tighter composition compared with conventional alloy 625, it exhibits excellent mechanical and fatigue properties.

In recent years, the increased use of alloy 625LCF in the temperature range of 593°C and 650°C for aerospace and other industrial applications has required more attention to microstructural and mechanical properties with extended exposures within this temperature regime. This is crucial in that alloy 625LCF, similar to alloy 625, has the potential to precipitate $M_{23}C_6$, M_6C, γ'' and γ' which can influence various mechanical properties such as impact and tensile strength and fatigue resistance. Also, extended exposure times could coalesce these precipitates, thereby further altering material performance.

Extensive characterization of conventional alloy 625 in this temperature regime has been done.[2] But there has been limited work done on characterizing the microstructural and mechanical stability of alloy 625LCF. Figures 1 and 2 are historical time-temperature-transformation (TTT) diagrams for solution treated alloy 625.[3,4] The TTT diagrams differ with respect to the precipitation of $M_{23}C_6$ and M_6C. This difference could be related to composition and grain size. The silicon and carbon content appears to have an effect on the quantity and type of carbides that are precipitated[5].

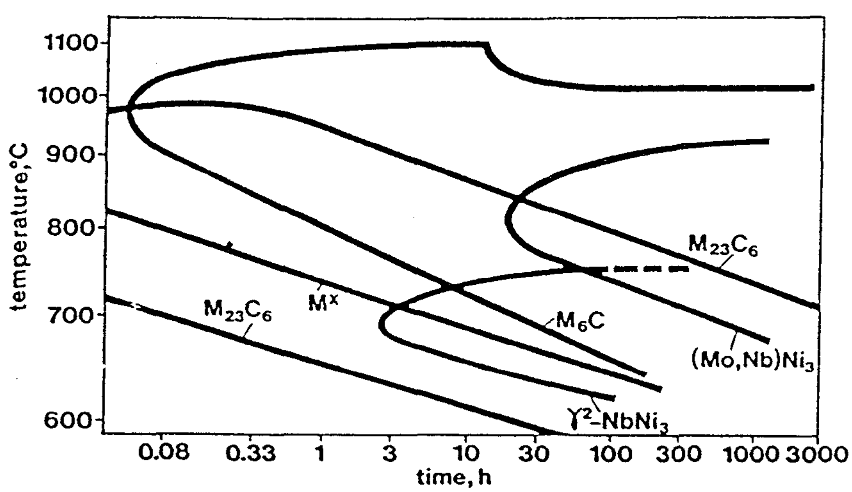

Figure 1 - Time-Temperature-Transformation Diagram for Solution Treated alloy 625, containing 0.37% Si. (Ref. 3)

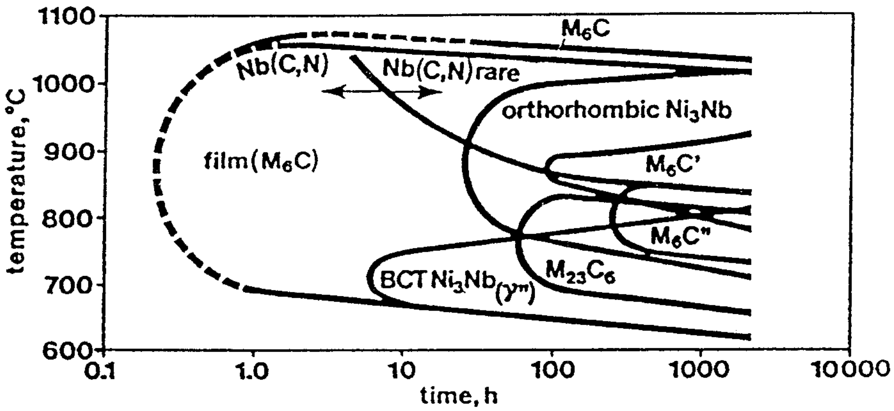

Figure 2 - Time-Temperature-Transformation Diagram of Solution Treated alloy 625, containing 0.22% Si. (Ref. 4)

Earlier studies of alloy 625 reported an abundant amount of γ'' in the matrix and δ (Ni_3Cb) in the grain boundaries with extended exposure at 650°C.[2] Also, it was determined that α-Cr formed at the grain boundaries and that it tended to increase with time.

It is the objective of this work to characterize the microstructural and mechanical relationship for extended times at 593°C and 650°C for alloy 625LCF.

Development of INCONEL alloy 625LCF

Alloy 625 sheet and strip is often manufactured using electric arc furnace melting, argon-oxygen-decarburization (AOD) refining followed by electroslag remelting (ESR). The cold rolled product is given a final hydrogen anneal at 1175°C which results in a product with a 0.2% yield strength of 330 to 380 MPa and an ASTM grain size number of 5 or finer. The extracted total residue contents (precipitates in the as-annealed condition) are shown in Table I. To reduce inclusion content, a VIM + ESR heat of alloy 625 was produced and processed with restricted carbon, nitrogen and silicon using the same strip manufacturing procedure employed for the AOD + ESR heat[6].

Table I: Precipitated Phases in Annealed INCONEL alloy 625LCF

Melt Method	Total Residue (weight %)	(Nb,Ti)(C,N) (weight %)	TiN (weight %)
AOD + ESR	0.38	0.30	0.08
VIM + ESR	0.09	0.06	0.03

In contrast to AOD + ESR produced alloy 625 it is clear that a vacuum induction melted (VIM) plus ESR of higher purity has a lower total residue content, particularly of (Nb,Ti)(C,N). This higher purity results in higher fatigue properties. See Figure 3.

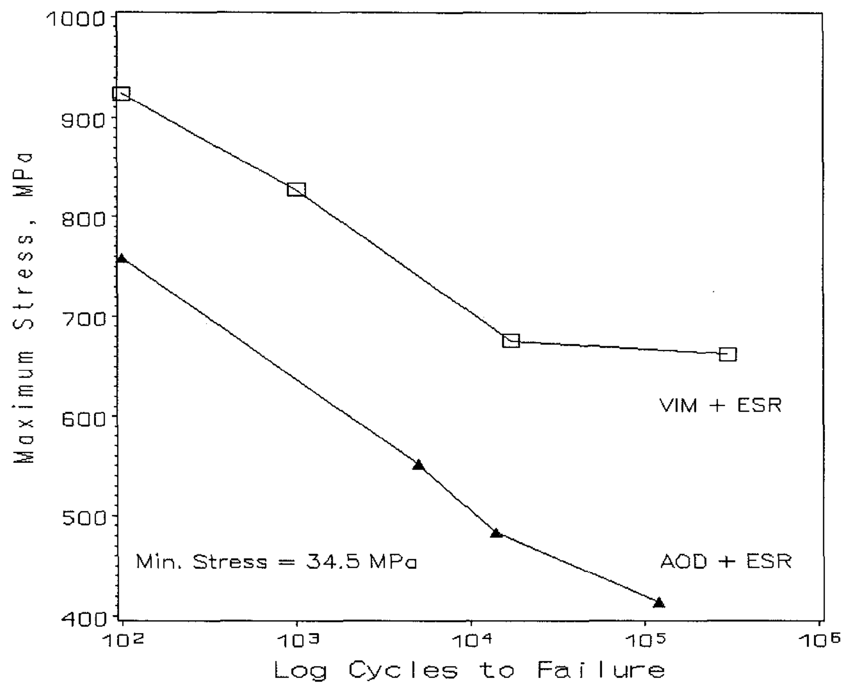

Figure 3: **Stress Controlled Tension-Tension Fatigue Properties at 540°C for Two Heats of Differing Composition and Method of Manufacture. (Ref. 6)**

Procedure

25.4mm thick alloy 625LCF plate and 1.02mm thick sheet, vacuum induction melted (VIM) plus electroslag remelted (ESR), were selected for the characterization study. Their compositions, as well as the nominal composition for alloy 625, are listed in Table II. The plate was annealed in air at 1010°C / 1.0 hr. The sheet was continuous annealed in hydrogen at 1024°C.

To characterize the effect of exposure at 593°C and 650°C, specimens were aged at each of these temperatures for various times up to 7500 hrs (11 months). Each condition was tested for room and high temperature tensile properties (ASTM E-8). The microstructures were analyzed via use of X-ray diffraction (XRD) of extracted phases and scanning electron microscopy (SEM) of the microstructure.

The metallographic samples were prepared by electro-polishing for 20 seconds at 25 volts in a 20% H_2SO_4-methanol solution after grinding them to a 6μm diamond finish. The samples were etched by electro-etching for 8 seconds in a CrO_3-H_3PO_4-H_2SO_4 solution at 5 volts. This etching procedure will put γ',γ'', and δ in relief to provide optimum contrast on the SEM.

The extractions were performed using 10% HCl-methanol at 5 volts for 1 hr with the residue ultrasonically removed in alcohol every 10 minutes. Part of the residue was applied to a carbon stub for SEM and EDS evaluation while the bulk of the residue was used for XRD studies to identify the phases. Because certain phases are not extracted by the etchant, the partially extracted

Table II - Weight Percent Compositions

Element	UNS N06625 Alloy 625*	Alloy 625LCF (Plate) 25.4mm Thick	Alloy 625LCF (Sheet) 1.02mm Thick
C	0.05	0.01	0.013
Mn	-	0.06	0.08
Fe	2.50	4.17	4.16
Si	0.25	0.08	0.08
Cu	-	0.08	0.07
Ni	61.0	60.9	60.4
Cr	21.5	21.8	22.0
Al	0.20	0.19	0.21
Ti	0.20	0.26	0.19
Mo	9.00	9.02	9.01
Nb	3.60	3.53	3.43

* Nominal Weight Percent Composition

sample was analyzed using SEM to determine phases remaining which were not removed during the extraction procedure.

Results

Mechanical property test results for alloy 625LCF after short term exposures for the sheet product and long term exposures for the plate product are listed in Tables III thru VI. Selected SEM/EDX photomicrographs illustrating the morphology of the various phases are shown in Figures 4 thru 9.

Mechanical Properties

The results listed in Table III are for short term exposures on alloy 625LCF sheet. The results show an increase in yield strength after short term exposures of approximately 24 hrs at 593°C and 16 hrs at 650°C.

The room and high temperature tensile properties of alloy 625LCF plate with extended exposure times at 593°C and 650°C reveal a reduction in ductility. See Tables IV and V. But the ductility remains above 20%. The yield and tensile strength results show a reduction after 5000 hrs (7 months) at 650°C and a steady increase up to 7500 hrs (11 months) exposures at 593°C. When long-term aging at 593°C and 650°C occurs, the original properties can be restored by annealing at 954°C/1 hr/AC. See Table VI.

Table III - Room Temperature Tensile Properties of Alloy 625LCF Sheet, After Aging at 593°C and 650°C for Various Exposure Times.

Aging Temperature	Aging Time (hrs)	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
As Annealed (1024°C in H_2)		525.4	941.2	46.5
593°C	1	544.0	951.5	46.4
	2	559.2	970.8	45.6
	4	559.9	969.4	45.1
	8	-	-	-
	16	568.1	970.8	45.5
	24	583.3	977.7	44.8
650°C	1	559.2	983.2	45.8
	2	542.6	943.9	45.9
	4	573.0	984.6	45.2
	8	591.6	985.3	42.9
	16	679.2	1,050.8	39.6
	24	756.4	1,123.9	37.6

Table IV - Room Temperature Tensile Properties of Alloy 625LCF Plate, After Aging at 593°C and 650°C for Various Times.

Exposure Time	Aging Temperature	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
As Annealed (1010°C in Air)		484.7	943.9	46.0
2500 hrs	593°C	890.1	1,257.0	29.0
5000 hrs		935.0	1,296.3	27.3
7500 hrs		1,029.4	1,341.8	22.3
2500 hrs	650°C	1,026.7	1,317.6	26.1
5000 hrs		972.2	1,287.3	21.0
7500 hrs		919.1	1,285.9	20.4

Note: Average of Two Specimens.

Table V - High Temperature Tensile Properties of Alloy 625LCF Plate, After Aging at 593°C and 650°C for Various Times. (The Test Temperature was the Aging Temperature).

Exposure Time	Age & Test Temperature	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
2500 hrs	593°C	734.3	1,050.1	29.5
5000 hrs		839.1	1,143.2	26.9
7500 hrs		906.7	1,183.2	22.2
2500 hrs	650°C	808.8	1,065.3	32.3
5000 hrs		809.5	1,080.4	24.7
7500 hrs		766.0	1,033.6	18.1

Note: Average of Two Specimens

Table VI - Room Temperature Tensile Properties of Alloy 625LCF Plate, After Aging at 593°C and 650°C for Various Times followed by an Anneal at 954°C.

Exposure Time	Aging Temperature	Yield Strength (MPa)	Tensile Strength (MPa)	Elongation (%)
As Annealed (1010°C in Air)		484.7	943.9	46.0
2500 hrs	593°C	450.9	913.6	52.4
5000 hrs		508.2	959.8	44.5
7500 hrs		482.6	947.4	48.6
2500 hrs	650°C	473.7	963.9	49.3
5000 hrs		503.3	969.4	45.1
7500 hrs		489.5	956.3	48.5

Note: Average of Two Specimens.

Microstructural Analysis

XRD and SEM evaluations revealed finely dispersed γ'' and γ' within the matrix after 24 hrs at 650°C and 7 months at 593°C. Extended exposure times ultimately transformed a portion of these finely dispersed precipitates to δ. This transformation was more pronounced at the grain boundaries. See Figures 4 - 7.

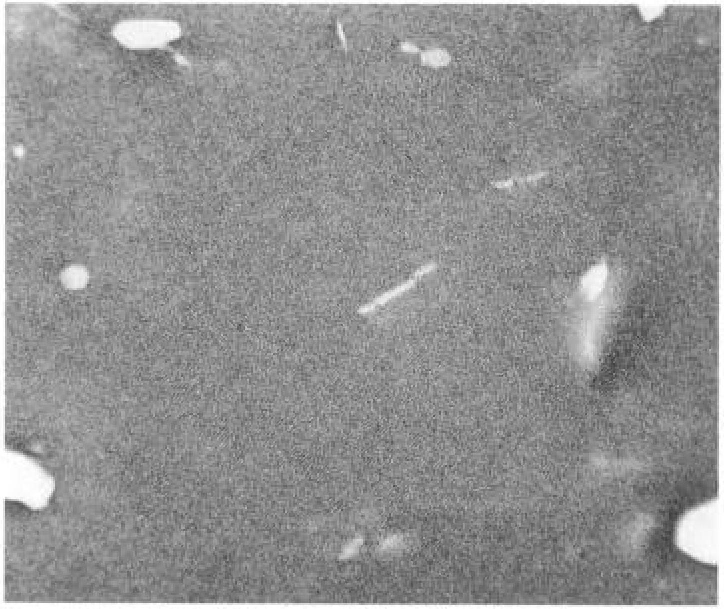

Figure 4 - SEM photomicrograph of alloy 625LCF after an exposure at 593°C for 5000 hrs (7 months). This illustrates the beginning formation of carbide plates and small quantity of γ'' and γ'.
Magnification: 10,000X

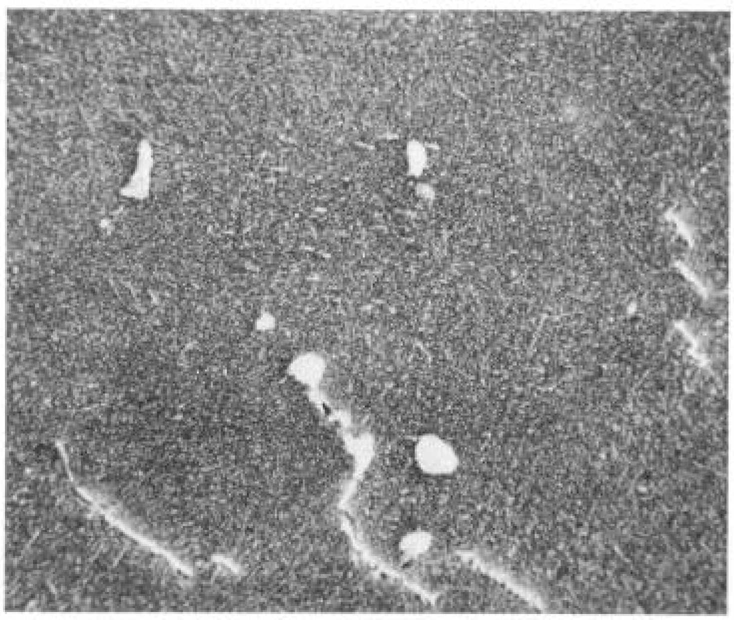

Figure 5 - SEM photomicrograph of alloy 625LCF after exposure at 593°C for 7500 hrs (11 months). This shows a larger amount of γ'' as well as some M_6C and MC carbides.
Magnification: 10,000X

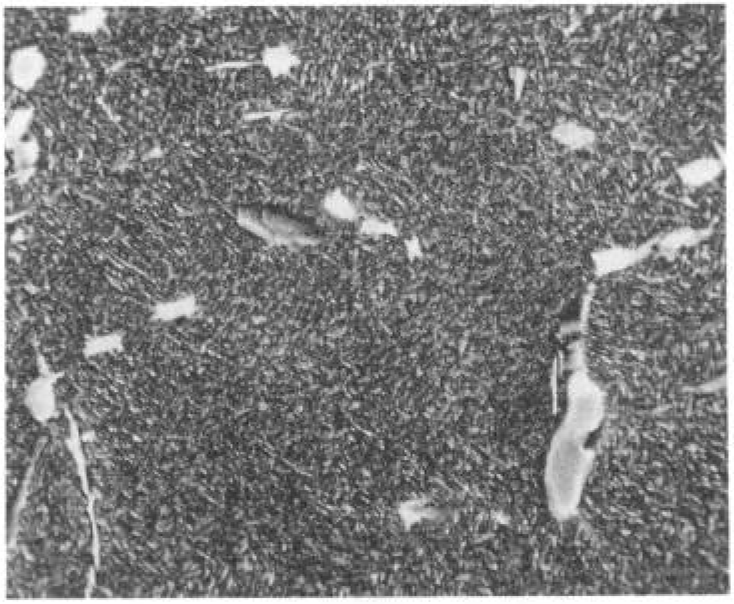

Figure 6 - SEM photomicrograph of alloy 625LCF after an exposure at 650°C for 2500 hrs (4 months). There are large amounts of γ'' similar to that of 593°C for 7500 hrs (11 months).
Magnification: 10,000X

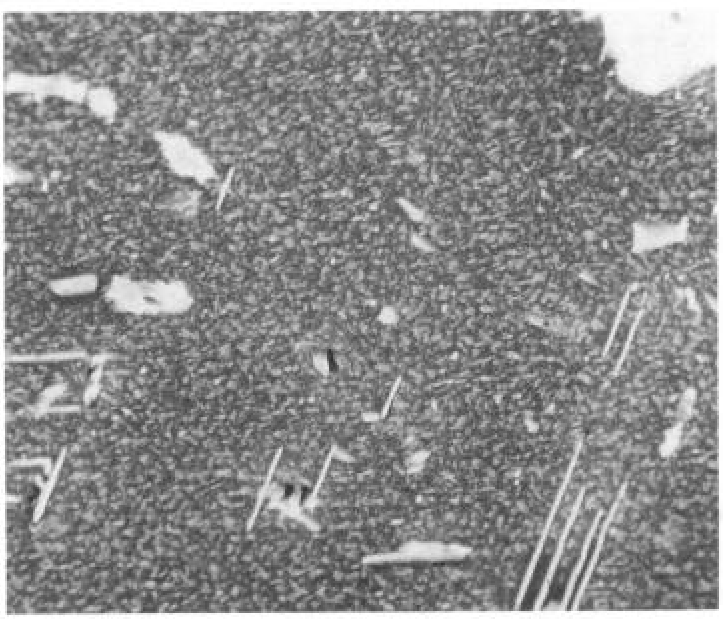

Figure 7 - SEM photomicrograph of alloy 625LCF after exposure at 650°C for 5000 hrs (7 months). There is some coarsening of carbides and a larger quantity of δ platelets forming.
Magnification: 10,000X

As in the case of alloy 625 it was found that alloy 625LCF precipitated α-Cr, Figures 8a and 8b. α-Cr was detected after approximately 2500 hrs (4 months) at 650°C and 7500 hrs (11 months) at 593°C. The α-Cr and δ most likely contribute to the loss in ductility after extended times at 593°C and 650°C.

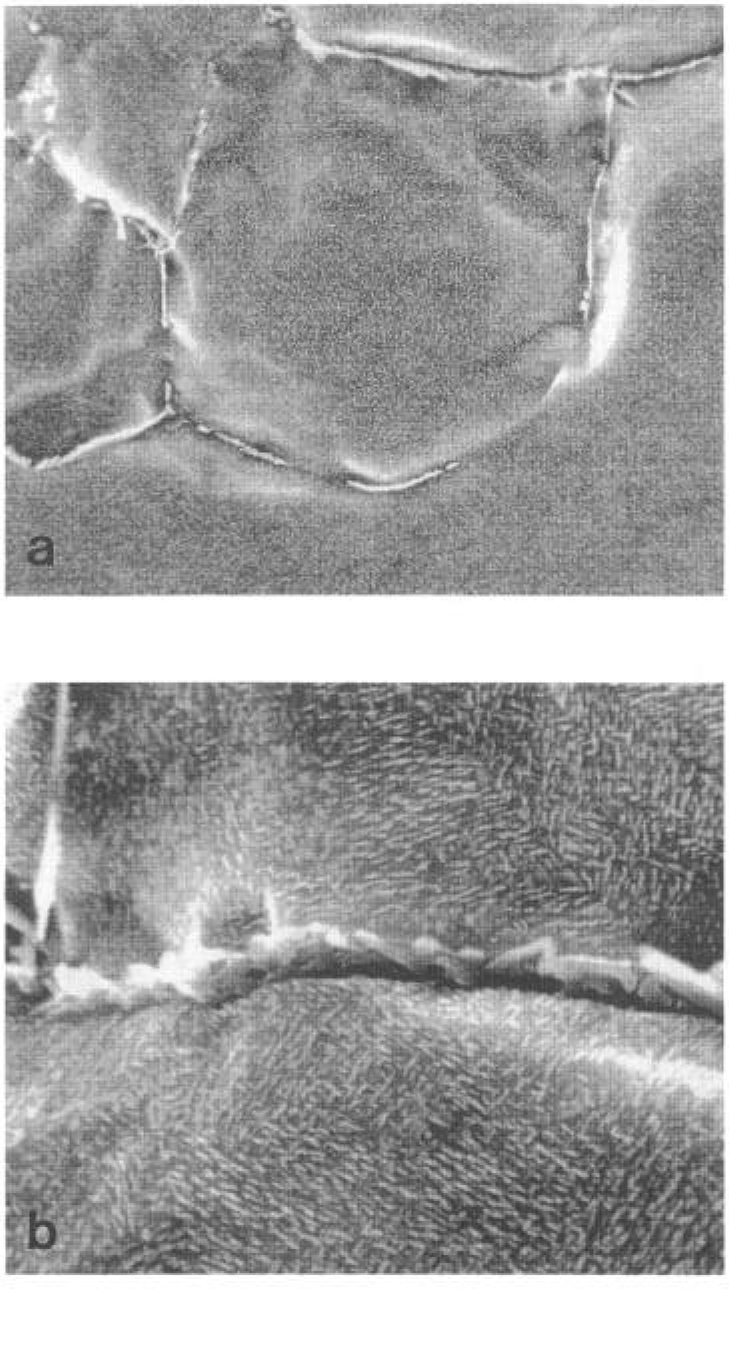

Figure 8 - SEM photomicrograph of alloy 625LCF after an exposure at 650°C for 7500 hrs (11 months). a) α-Cr is associated with the grain boundary b) A higher magnification of the grain boundary. Magnification: a) 3,000X b) 10,000X

In contrast to results of previous studies of alloy 625, the XRD did not detect any $M_{23}C_6$ carbide in alloy 625LCF. As mentioned previously, the most likely reason for this would be the low carbon and silicon content of alloy 625LCF. M_6C and MC carbide precipitates were detected. It is considered that these carbides are introduced during the anneal, Figure 9.

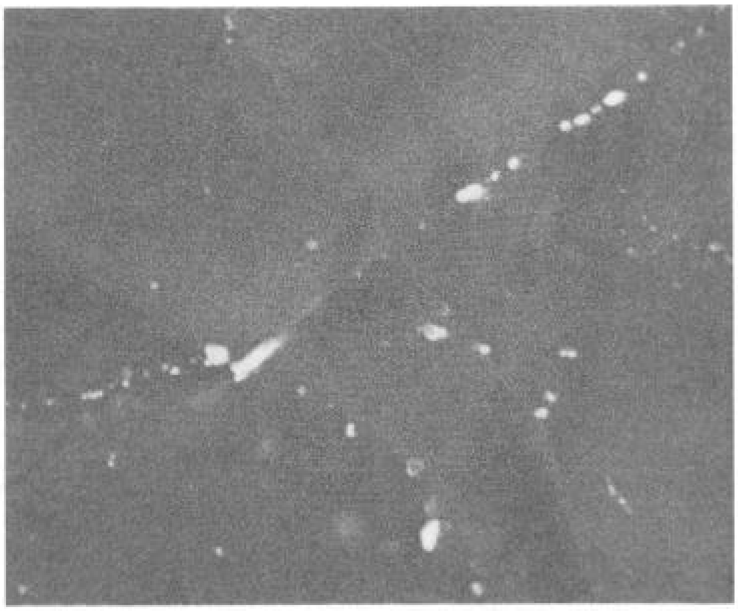

Figure 9 - SEM photomicrograph of As-Annealed alloy 625LCF. There are finely dispersed M_6C and MC carbides introduced by the 1010°C anneal.
Magnification: 10,000X

Understanding of the long term stability of alloy 625LCF would be enhanced with an extension of this study to 820°C with additional characterization of phase growth kinetics and phase volume percentage as a function of time and temperature.

Conclusions

1) XRD analysis of alloy 625LCF at 593°C and 650°C after extended exposures up to 7500 hrs (11 months) did not detect $M_{23}C_6$ in the microstructure.

2) Both extractions and SEM evaluations confirm that extended exposure for 7500 hrs (11 months) at 593°C and 2500 hrs (4 months) at 650°C precipitated α-Cr at the grain boundaries.

3) XRD and SEM evaluations revealed finely dispersed intragranular γ'' and γ' after 24 hrs at 650°C and 5000 hrs (7 months) at 593°C. Longer exposure times exhibited larger quantities of these phases with a portion ultimately transforming to δ phase. The tendency for this transformation was more pronounced at the grain boundaries.

4) M_6C and MC carbides are introduced during the anneal of alloy 625LCF.

5) Extended exposure on room temperature yield strength at 593°C and 650°C shows an increase in yield strength at 593°C for exposures up to 7500 hrs (11 months), but decreases after 5000 hrs (7 months) at 650°C.

6) After extended times at 593°C and 650°C there is a reduction in tensile elongation. But it remains greater than 20%.

7) An anneal of 954°C/1 hr/AC after all exposures at 593°C and 650°C restores alloy 625LCF original properties by re-solutioning α-Cr , δ and γ''.

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