#### SEGREGATION OF DEFECTS IN WROUGHT ALLOY 718

#### MANAGEMENT OF INDUSTRIAL SAFEGUARDS

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#### ABSTRACT

Alloy 718 is largerly used for jet engine critical parts manufacturing. This type of application needs a carefull survey of melting and forging steps of fabrication.

This alloy is difficult to melt due to its particular properties and its sensitivity to potential process instabilities.

The melting processes are briefly reviewed according to these difficulties. The main inspection methods are compared and detectable segregations are characterized in terms of chemical composition.

Improvements of the process have significantly decreased the risk of freckles. White spots are always a concern: their origins are analyzed, as well as the their effects on mechanical properties. The inspection procedures devoted to minimize this defect occurence are exposed.

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#### 1 - Introduction

The production of high quality Alloy 718 faces two fundamental melting process obstacles which are: 1/ the solidification temperature interval and 2/ the heavy element segregation which accompanies this solidification.

## Physical-chemistry of Alloy 718

Solidus and liquidus temperatures are respectively 1260 °C and 1335 °C, which makes a significantly larger scatter than for many other nickel base alloys (see table I). As a result, the volume of the liquid metal phase is more voluminous during remelting and prone to convective flow. According to the multi-phase diagram, it is enriched in heavy Niobium element. Thus, this alloy has a tendency to give freckles, due to the collapse of the solidification line and drop of high Niobium content material between the dendrites.

From an industrial point of view, the problem of the liquid mass has restricted the melt of high quality Alloy 718 below a 500 mm (20 inches) diameter.

Table I - S	Solidus	and L	iauidus	Temperatures	of	Various	Nickel	Base Alloys
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Alloy des	Alloy designation		Temperature (°C)		
AFNOR	Commercial	Solidus	Liquidus	(LiqSol.)°C	
NC15Fe	Inco 600	1370	1420	50	
NC19FeNb	Inco 718	1260	1335	75	
NC20K14	Waspaloy	1330	1360	30	
NK15CATu	IN 100	1230	1310	80	
NK17CDAT	Astroloy	1263	1330	67	
NC22DNb	Inco 625	1275	1350	75	
NC18KTDAW	Udimet 720	1240	1335	95	

## Remelting process stability

The remelting by electrical means consists of providing a high density energy on a point by point basis. It is important that overheats followed by solidification sequences are prevented, thus:

- local temperature variations versus time have to be avoided and in every case much lower than the solidus-liquidus scatter,
- the melting process may not be stopped, i.e. the residual pressure has to be low enough to prevent the glowing discharges during which the phenomenon is stopped if the electrical power it not sufficient. Segregation occurs when the melt is started again.

Furthermore, slight changes in electrical intensity result in dangerous variations of the liquid depth as it has been evinced by F.J. ZANNER [1].

Based upon the above remarks, a review of the current melting and refining processes is proposed hereunder.

# 2 - Nickel base alloy melting

The process consists of :

- a first melt made in a  $\underline{V}$  acuum  $\underline{I}$  nduction  $\underline{M}$ elting furnace.
- one or two remelts in <u>Vacuum Arc Remelting furnace</u>.
- possibly, one Electro Slag Remelting between VIM and VAR.

## Vacuum Induction Melting

The first melting allows to get an accurate chemical composition and a low value of both carbon and oxygen contents. This will prevent the growth of large carbides and will enhance the low cycle fatigue properties. Other elements as  $H_2$ ,  $N_2$  etc. and trace elements are also minimized.

Some parameters are to be continuously recorded:

- residual pressure over the molten metal,
- electrical energy consumption along the melting,
- temperature before pouring.

Nevertheless, as low as it can be, the nitrogen content (currently 70 ppm) is high enough, owing to the Nb and Ti concentrations, to promote nitride precipitation in the liquid bath.

#### Remelting processes

The purposes of the remelting processes are :

- deletion of macro segregations and porosities,
- reduction of micro segregations,
- hydrogen minimization.

Such a process results in improvements of mechanical properties [1] :

- fatigue properties because of inclusion reduction, sulphides and oxides,
- fracture toughness.

An other point is that the mechanical properties with regard to the direction of specimen cut inside the ingot are very close.

In the VAR furnace, the melt is obtained by a high intensity electrical current initiated between the electrode and lower side of the mold made of copper. The ESR process also operates under high intensity electrical power melting by Joule effect with a CaO/Al<sub>2</sub>O<sub>3</sub>/CaF<sub>2</sub> basic slag through which the liquid metal is transferred and refined, as explained by JAEGER, HOCH, CERWENKA [3].

The liquid metal pool depth and the risk of freckles are directly associated. In the VAR process, this event has been early experimented. The use of a helium flow in the mold has resulted in a high heat

extraction rate and minimizes the pool depth. In counterpart, the basically instable remelting conditions have associated the risk of white spot occurence to the VAR process. During ESR the slag acts as a thermal insulator resulting in a comparatively higher depth of liquid metal prone to freckling, but the thermal inertia minimizes the white spot occurence.

However, recent improvements may have been brought due to a better know-ledge and control of the ESR process (electrode location in the slag and electrical connection at the bottom) and it is possible to avoid the freckle defect on an industrial basis in ingots of 450 mm in diameter.

As of today, the VIM-VAR process is widely used for rotating parts, but it will be shown below that the triple melt VIM-ESR-VAR obviously produces a more reliable quality of 718.

## 3 - Most commonly observed defects and methods of inspection

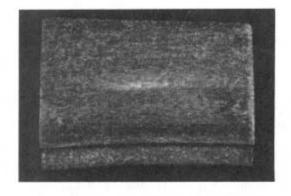
## 3.1 - Defect identification

Engine manufacturer, AMS or AECMA standards give an accurate definition of the defects.

White spots are white areas which appear after an appropriate etch. They are extended areas depleted in metallic hardening elements, typically Nb, Ti or Al in 718, and may appear as "fibrous" or "bulk" as shown on the figure 1. They are often surrounded by hard particles such as oxides or carbonitrides.

<u>Freckles</u> appear as black after etching, are enriched in the same elements segregating out of the white spots and currently contain brittle precipitates which are the ultimate step of the alloy evolution under ageing.

<u>Tree rings</u>: they are annular type contrasts created by the sudden change of dendrite orientations due to rapid differences in the pool depth encountered in the bottom of the ingots and are witnesses of slight melting instabilities which may result locally in white spots and freckles. This phenomenon may also result locally in <u>Light Etching Areas</u> identified by the Cytemp Company some years ago as being from different origin than white spots.



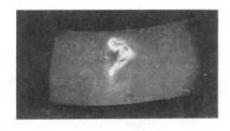


Figure 1 - Example of "fibrous" and "bulk" white spots

## 3.2 - Methods of inspection

## 3.2.1 - Macrographic inspection

Macrographic inspection is the most efficient method of segregation detection.

## The "Canada Etch"

The bath is a mix of water, sulfuric, hydrofluoric and nitric acids. It has a relatively quick effect (only a few minutes to reveal the "dendritic" segregations and the freckles). But it is difficult to use because of the hydrofluoric acid temperature of use (73 °C).

# The Electrolytical etch

Electrolysis of several dozens of Amperes in a diluted acid bath is utilized at SNECMA. This etch has proved its efficiency especially in detecting the interdendritic segregations.

#### RMAC 13

Among the different charts experimented by SNECMA, the most efficient is RMAC 13 which consists of :

- a bath made of ferric chloride (FeCl<sub>3</sub>) and hydrochloric acid in which a moisturizing agent may be added; immersion time of the component is approximately 10 minutes at a temperature of 45 °C. The bath is permanently agitated,
- the part is afterward rinsed and whitened in an agitated bath of nitric and hydrofluoric acid for at least one minute, at a temperature slightly above the room temperature. It is rinsed again, then dried. SNECMA's chain of control is automatized through a robotic system. A general view is shown on the figure 2.

There are other efficient etches but their use in industry is difficult because their performances decrease when ageing.

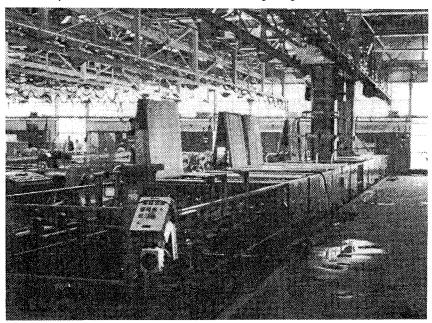


Figure 2 - General view of SNECMA automatized etching facilities

#### 3.2.2 - Ultrasonic inspection

Metallurgical defects have been detected by performing US inspections:

- in 60 % of the cases, the defect is a white spot with peripherical decohesions on clusters of carbonitrides rich in Nb and Ti, and oxides

rich in Al and Mg. There are almost no cases of U.S. detected white spots without any decohesion.

- in the other cases, indication cut-ups do not show any chemical segregation, but they reveal clusters of carbonitrides.

It is always a concern that US indications may be due to decohesions surrounding dirty white spots. This has been pointed out in particular by CREMISIO [4].

# 3.2.3 - Correlation between etch indications and shift of the chemical composition

In order to know the chemical composition shift accompanying the dendritic segregation detected by etching specimens, a laboratory study was conducted by using a Scanning Electron Microscope (SEM) equiped with a wave length spectrometric tool [2], so that the accuracy of the measurement is less than 0.1 % in weight with a spatial resolution of some microns. In addition, the tests were performed with specimens having sustained one or two homogeneisation heat treatments.

Test conditions and results of the experiments are as follow:

- a first set of samples has sustained one single homogeneisation heat treatment after the VAR remelting of a 500 mm diameter ingot: the 1180 °C temperature was maintained during 24 hours on the  $\varnothing$  200 mm billet,
- a second set of samples was etched after the following process :
  - . 1150 °C during 24 hours of the 500 mm diameter ingot,
  - . forging down to 330 mm diameter,
  - . 1180 °C during 72 hours,
  - . forging down to 200 mm diameter.

All specimens were heat treated at 1040 °C during 1 hour in a way to solution the  $\delta$  phase and delete the effect of the cold drawing.

After having identified by etching representative areas of standard dendritic spacing, the chemical fluctuations have been evaluated by Scanning Electron Microscope (SEM).

The table II shows that:

- segregations are affecting iron, chromium and niobium,
- a total shift by 0.015 % in weight of the three above elements can be detected by micrography,
- as shown on Table II, the decrease in chemical composition shift due to a second homogeneisation heat treatment at a former step of the process are as follow:
  - . for iron, the initial gap is divided by a factor 5.
  - . for chromium and niobium this factor is between 10 and 20.

It must be added that this segregation softening is highly dependent of the dendrite size and fully inoperant at the centre of a defect larger than 1 mm.

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These data point out the etching method sensibility, the efficiency of the soaking process and the inability of this technique to solve the segregation problems attached to largely extended defects such as white spots and freckles.

Table II - Chemical Segregation after Different Homogeneisation Cycles

Homogeneisation cycle	Specimen number	ΔFe (%)	ΔCr (%)	ΔNb (%)
	1	0.90	0.45	- 0.35
1180°C - 24 hours	2	0.90	0.90	- 0.55
on ø 200 mm billet	3	0.90	0.80	- 0.90
On ø 500 mm ingot:	4	- 0.026	- 0.085	0.075
1150°C - 24 hours	5	0.056	0.066	0.013
+	6	0.164	0.003	0.051
On ø 330 mm billet:	. 7	0.201	0.029	- 0.026
1180°C - 72 hours	8	- 0.029	- 0.041	0.08

# 4 - Potential origin of white spots in Alloy 718

We have summarized above the prior difficulties attached to 718 processing, thus many parameters have been identified as causes of white spots. In fact, each instability of the melting process may result in segregations.

The fundamental objectives of the VAR process are to:

#### 4.1 - <u>Insure good power distribution and heat transfert</u>

- An uniform distribution of the electrical power, which may be reached only if the assembly of the stub and the electrode in the mold is axisymetric. The cooling devices by helium and water circuits are also important to keep a smooth and uniform liquid pool.
- Electrode assembly: melters have their own procedure to insure a good assembly: it is obvious that the displacement of electrode during the remelting has been correlated to US indications corresponding to time as long as 30 minutes after the incident.

#### 4.2 - Insure a steady state remelting condition

The melters have to keep a good control of the gap between the electrode and the liquid pool. This parameter is not directly measured and has to be estimated from electrical signals attached to the arc behaviour.

During the early years of the VAR process, the voltage was considered but was not sensitive enough. "State of the art" furnaces operate with hash count or drip short concepts and the measurement of the melt rate is only a help to initiate the melt.

- Cooling system : the helium circuit makes the heat transfer from electrode to the water circuit more easy. In case of failure, the heat

extraction is disrupted and pool depth increases. The helium flow, the waterflow and inlet/outlet temperature should be permanently monitored.

The electrical parameters sometimes do no work properly because of adverse effects related to abnormal electrode melting.

Pipe in the electrode is very detrimental due to two kinds of problems :

- first, the arc may focus inside the pipe but the outer diameter of the electrode does no more melt. The cavity volume is increased by such melting erring ways and pieces of electrode fall from the pipe into the pool,
- if the pipe is full of gas, as carbon oxide or hydrogen, the change of the dielectric properties of the gap may give a glowing discharge. In that case, the melt is stopped and the controls hold up the electrode.

In both cases, an erratic up-down movement of the electrode may occur and its surface temperature may go through solidus and liquidus lines, resulting in severe surface segregations. During the time this discrepancy occurs, the relative distance from the electrode to the pool and mold changes in such a range that an arc ignition on the crown may break solid pieces of material.

The origin of above described defects can be identified according to some analysis, as recommended by EVANS, RADAVICH [5] and MITCHELL [7]:

- classic white spots are either due to bulk fall-in from the electrode showing concentrations of magnesium or condensate falls due to arc erratic movement, characterized by copper traces. These two defects exhibit a lower hardness than the matrix. There is generally no  $\delta$  phase precipitation because of the very low niobium content,
- "solidification white spots" are associated with dendritic aggregates accompanying tree-rings. They are not so poor in niobium and the hardness drop is only 0 to 3  $R_{\text{\tiny C}}.$

## 5 - <u>In-depth inspection of a typical defect</u>

A typical fall-in defect has been inspected by CHORON and MARTIN [6]. Parameters of the ultrasonic investigation are the following: 150 mm diameter billet, frequency: 5 MHz, longitudinal waves. The ultrasonic indication is equivalent to that of a 0.8 mm flat bottom hole. It is located at 75 mm depth from the surface.

First, the material is cut at about 70 mm from the surface and then it is polished on about 15 cuts through the defect. The main observations are :

- a pronounced dendritic structure with a drop in hardness of about 70 HV points compared to the matrix,
- classical white areas surrounded by oxides rich in Aluminum, Titanium, Magnesium and Titanium carbonitrides (figure 3). The grain size varies between ASTM 3 in the defect and ASTM 6 in the matrix. The hardness falls down to 350 HV in the white spot,
- 5 mm from the ultrasonic defect, hardness is only at an average rate of 310 HV with a point at 280 HV but the variations in chemical compositions are very small,

- at the location of the ultrasonic defect, the segregation extent measures 12  $\,\mathrm{mm}^2$  and is limited by a decohesion of 0.2  $\,\mathrm{mm}$  large and 3.5  $\,\mathrm{mm}$  in depth.

Such a defect comes from the fall in of a solid piece of electrode which has been surrounded by floating inclusions. Only the decohesion has made the ultrasonic detection possible.

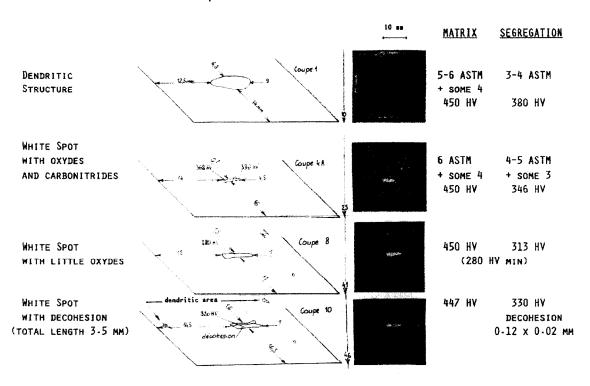


Figure 3 - White spot characterization

#### 6 - Effect of white spots on mechanical characteristics

#### 6.1 - Tensile test

Tensile tests have been performed on specimens at room temperature. It appears from the table III that the values are quite acceptable except for some result as low as Y.S. = 829 MPa.

Table III - Effect of white spots on tensile properties

	Tensile properties							
Sampling	(MPa)	1	2	3	4	5	6	
Specimen taken in a white spot zone	Tensile strength	1131	1372	1269	1369	1364	1401	
	Yield strength	829	1137	1091	1155	1161	1128	
Specimen taken in a clear adjacent zone	Tensile strength	1350	1384	-	1410	1410	-	
	Yield strength	1222	1244	-	1202	1202	-	

# 6.2 - Fatigue test under strain or stress control

Low cycle fatigue with strain control R $\epsilon$  = 0 at 350 °C and  $\Delta\epsilon$ lt % = 0.56 have been made on samples taken from a turbine disc.

The samples have been machined in such a way that the white spot is located in the effective section of the sample. The results obtained are reported on figure 4: the drop in acceptable stress level is about 30% in comparison with the reference data.

This occurs regardless of what the percentage of white spots is in the sample surface. The reference samples show a homogeneous microstructure with thin grain (ASTM 10), while the "white spot samples" show a thin microstructure (ASTM 10) and a coarser one in the defected area (ASTM 5 to 6).

The same kind of tests has been performed under stress control condition:  $\Delta \sigma = 1130$  MPa,  $\theta = 550$  °C (see figure 5). Due to the fact that both strain/stress test are made within the elastic behaviour area of the material, the results are quite the same, but it is very important to observe that some specimens have failed during the first loading step.

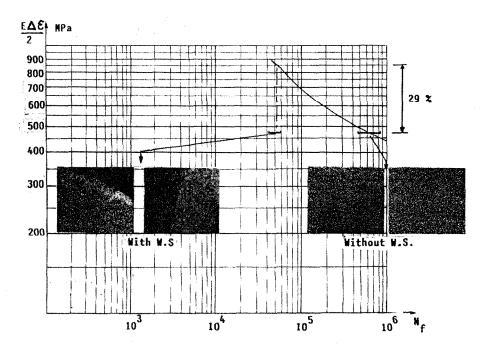


Figure 4 - Influence of white spots on strain controlled low cycle fatique

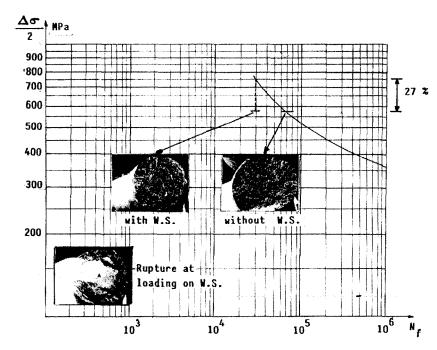


Figure 5 - Influence of white spots on load controlled low cycle fatigue

## 7 - Discussion and concluding remarks

All above exposed concerns summarize a long past experience of SNECMA Engineering and Quality Control organisations.

The Alloy 718 is widely used to manufacture jet engine discs because of:

- its mechanical performances which are very high at temperatures up to 550 °C-600 °C, with lives up to thousands of flight cycles,
- furthermore, its rupture-elongation at high temperature which makes it highly reliable.

These attractive properties are accompanied by a good forgeability. Furthermore, the absence of cobalt, which potentially minimizes an historic parameter of significant cost fluctuation, contributes to the interest for this material.

For these above mentionned reasons, the demand for forging stock has increased in such a way that 718 billet is the most ordered superalloy by-product.

After having experienced the melting of Waspaloy during years, the melters had to face the problem of freckles in Alloy 718. This major difficulty has been solved by process improvements including a lower melt rate.

As a consequence, the steady state conditions of the process are difficult to control and they are very sensitive to:

- electrode installation in the furnace,
- quality of the furnace, i.e. helium and water cooling circuits, electrical power distribution,

 electrode structure : bad surface roughness, gas pocket entrapped during its pouring, result in melting instabilities.

Each shift may result in white spots which are not always detected by non destructive tests but it is evinced that their effects on tensile and fatigue properties are unacceptable. High efficient etchings are used on an industrial basis in order to detect white spots at different steps of the disc machining process: associated with ultrasonic inspection, they almost cancel the risk of residual defect, at least in the thin parts.

State of the art consists of melting 500 mm diameter ingots which are converted to ASTM 5-6 billets.

80's requirements for new engines are about higher tensile properties resulting in higher Niobium content. Subsequently, the risk of defects is increased and new development of the process has been brought by the VIM-ESR-VAR route: it is obvious that the ESR step allows to reach a tightened chemical composition and a high quality electrode. The subsequent VAR refines the dendritic structure, minimizes the accompanying solidification segregations and lowers the nitro-carbide size distribution.

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