WHITE SPOTS IN SUPERALLOYS

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Abstract

A joint effort involving alloy producers, forgers, and users of vacuum melted nickel-base alloys under the auspices of ASM International has led to a common vocabulary for the different types of solute-lean defects found in superalloys; an improved understanding of their formation mechanisms, detection, and influence on mechanical properties has also been gained. These defects, more commonly known as white spots, have been classified into three types: discrete, dendritic, and solidification. Depending on factors such as size, chemistry, grain size, and the presence or absence of oxide/nitride clusters, discrete and dendritic white spots may be deleterious in highly stressed parts. Solidification white spots are generally not associated with non-metallic clusters and appear to have little effect on mechanical properties. However, additional low cycle fatigue results are needed. Proposed mechanisms of formation are different for each type of white spot.

Superalloys 718, 625, 706 and Various Derivatives
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Introduction

In April 1991, the first of three workshops was held to discuss ways in which the gas turbine industry could better understand defects in nickel-base superalloys. The workshops were sponsored by the Gas Turbine Superalloy Committee of the Aerospace Division of ASM International. Participants included representatives of metal producers, forgers, and engine manufacturers from five countries. The group's primary objective was to better define, and expand knowledge about, segregation in superalloys such as Alloy 718 and Waspaloy, with emphasis on light-etching areas referred to as solute-lean defects or "white spots." This "White Spots Committee" formed four subcommittees to focus efforts on classification, inspection, mechanisms, and mechanical properties.

A primary purpose of this article is to formalize the characterization and classification of white spots in high-strength superalloys so that the metallurgical community will use a common vocabulary when referring to them. An overview of formation mechanisms is presented along with preliminary test results, which should help shed light on the effects of solute-lean microstructures on tensile and fatigue properties. Also, a brief description of detection methods is provided. Although white spots are not limited to any single superalloy or class of superalloys, Alloy 718 is emphasized because it is so widely used, and because its relatively large solidus-liquidus temperature interval (~75°C, 135°F) and high niobium content (~5.3% Nb) make it prone to segregation.

Three distinct types of white spots have been identified and named by the committee: discrete, dendritic, and solidification white spots.

Classification

Discrete White Spots

A large discrete white spot in an Alloy 718 billet slice is shown in Figure 1. It appears bright and has a distinct boundary. Discrete white spots are usually located from the mid-radius to the center of the billet cross section. An Alloy 718 billet slice containing a small discrete white spot is shown in Figure 2. Depending on thermomechanical history, the grain size of a discrete white spot can be equivalent to or larger than the matrix grain size. Grain growth is illustrated in Figure 3, which shows a discrete white spot in an Alloy 718 forged part. A microhardness traverse of the white spot shown in Figure 3 revealed a hardness gradient from the matrix interface to the center of the white spot, which had the lowest hardness as shown in Figure 4.

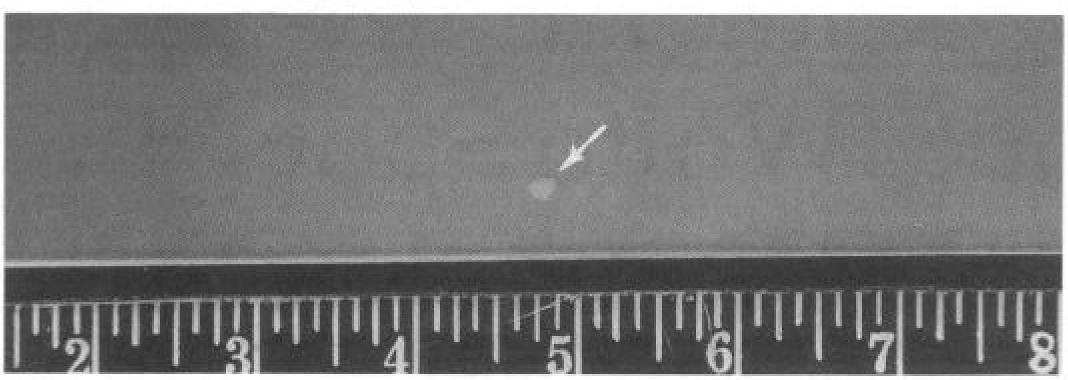


Figure 1. Large Discrete White Spot in an Alloy 718 Billet Slice. Canada Etch. Scale in Inches.

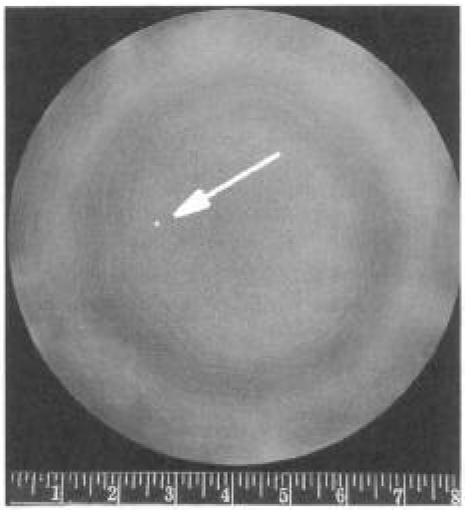


Figure 2. Small Discrete White Spot in an Alloy 718 Billet Slice. Canada Etch.

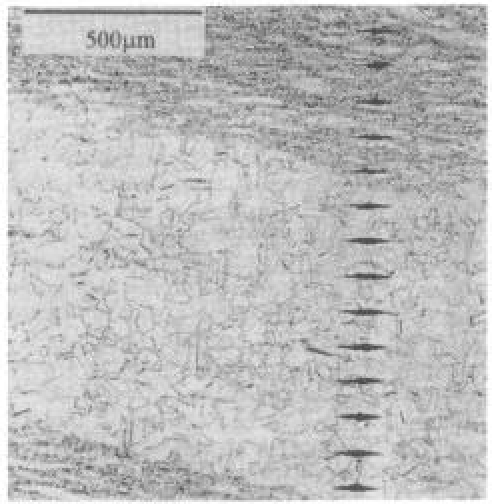


Figure 3. Discrete White Spot in an Alloy 718 Forging Showing Grain Growth. The Marks Traversing the White Spot in the Forged Part are Microhardness Indentations. Modified Kalling's Etch.

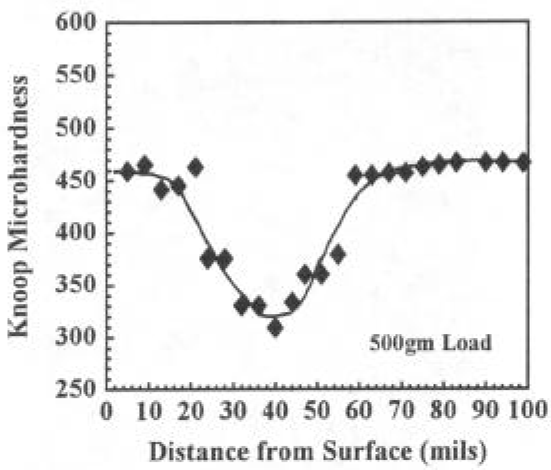


Figure 4. Microhardness Traverse of the White Spot Shown in Figure 3.

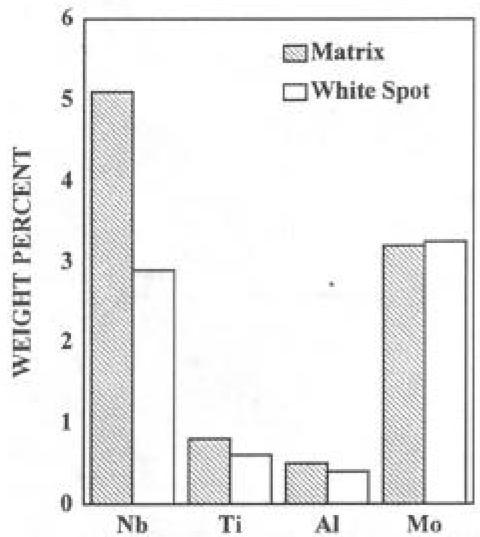


Figure 5. Energy-Dispersive X-Ray Spectroscopy (EDS) Analysis of the Discrete White Spot in the Alloy 718 Billet Slice Shown in Figure 1.

Scanning electron microscopy using energy- dispersive X-ray spectroscopy (EDS) found the white spot in Figure 1 to be depleted in niobium and, slightly, in titanium and aluminum (see Figure 5). In this example, the molybdenum level appears to be equivalent to that of the matrix. However, this element frequently is slightly depleted in discrete white spots. The white spot was enriched in nickel, chromium, and iron. The discrete white spot in Figure 2 exhibited less depletion as shown in Figure 6. These results are from three laboratories with different types of equipment ranging from EDS without standards to WDS (Wavelength Dispersive Spectroscopy) with standards and ZAF corrections. Results were normalized to the matrix chemistry. Agreement between laboratories is very good. Niobium depletion in Alloy 718 white spots has been shown to vary between -0.75 and -3%. For other alloys, element depletion usually is less severe, as shown by the EDS data in Figure 7 for a discrete white spot in Waspaloy.

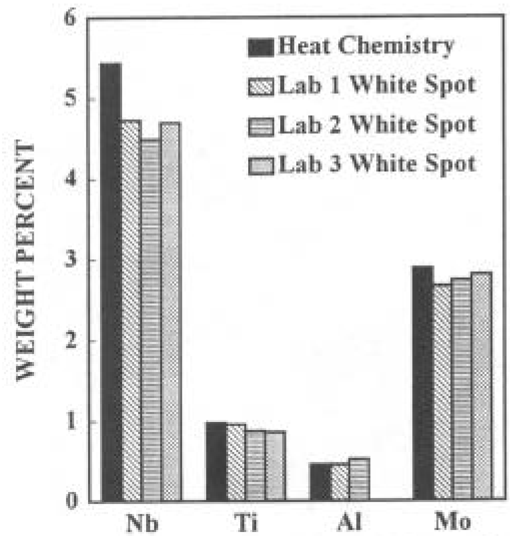


Figure 6. Analysis by Three Different Laboratories of the Discrete White Spot in the Alloy 718 Billet Slice Shown in Figure 2.

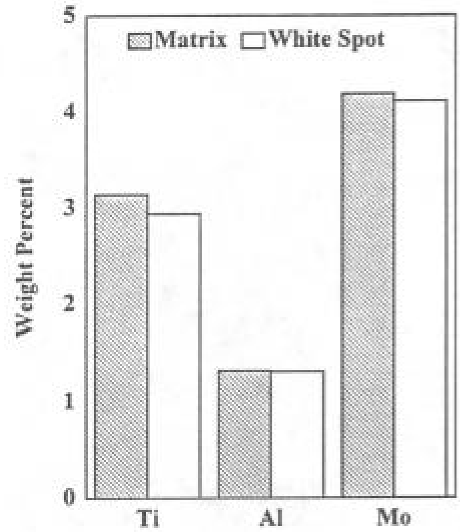


Figure 7. EDS Analysis of a Discrete White Spot in a Waspaloy Billet Slice. Element Depletion is Less Severe than for Alloy 718.

Figure 8. Unlike the Matrix (left half), a Discrete White Spot (right half), in a Waspaloy Billet Slice has no Precipitated Carbides. As Polished.

Another element that is always depleted in discrete white spots is carbon, as evidenced by a reduced density of carbides. An example is shown in Figure 8 for Waspaloy that had been processed in a manner that precipitated carbides at prior grain boundaries.The white spot is made apparent by the absence of precipitated carbides.

(a)

(b)

(c)

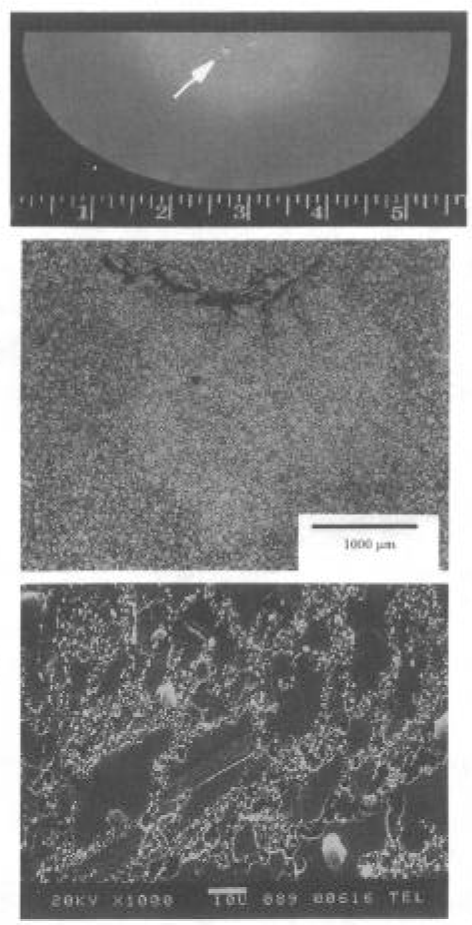


Figure 9. Dirty Discrete White Spot in an Alloy 718 Forged Part. (a) Macro, (b) Optical Micrograph Showing a Crack, and (c) SEM Micrograph Showing a Cluster of Inclusions.

Discrete white spots sometimes are associated with clusters of oxide, nitride, and/or carbonitride particles. These are termed "dirty" discrete white spots. If the cluster is sufficiently large, a crack may be present. Many ultrasonic indications in superalloys turn out to be cracks at clusters of inclusions associated with discrete white spots. An example is shown in Figure 9 for an Alloy 718 billet slice.

Dendritic White Spots

The Alloy 718 billet slice shown in Figure 10 contains one large and two small dendritic white spots. They have a dendritic appearance and a diffuse interface with the matrix. Dendritic white spots are located close to the center of the billet cross section. The dendritic appearance is illustrated in Figure 11, where macroetched dendritic and discrete white spots in Alloy 718 are compared at low magnification, and is further emphasized in Figure 12, which shows a cluster of two dendritic white spots in a longitudinal billet section of Alloy 718. (Dendritic white spots tend to appear in clusters.)

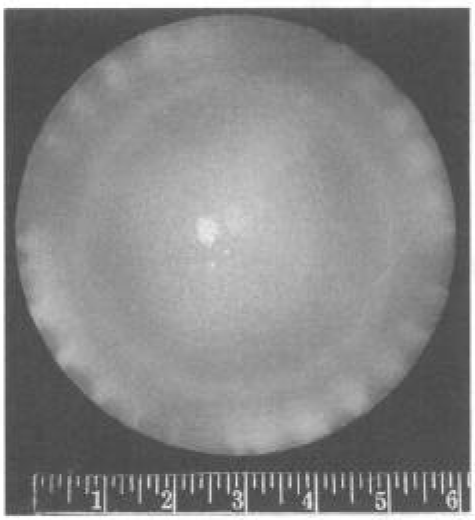
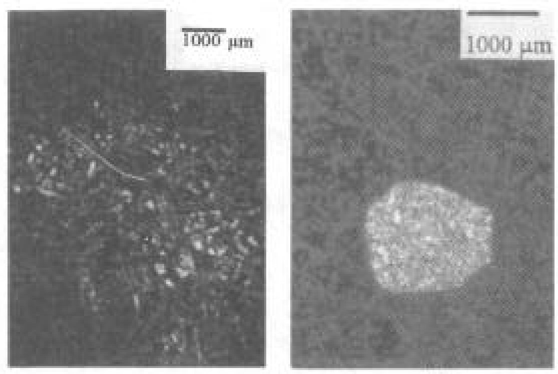


Figure 10. Dendritic White Spots in the Center Region of an Alloy 718 Billet Slice. Canada Etch.



(a) (b)

Figure 11. Comparison, at Low Magnification, of Dendritic (a) and Discrete (b) White Spots in Alloy 718 Billet Slices. Canada Etch.

EDS data for depleted elements in light areas, dark areas, and a large area of a dendritic white spot are shown in Figure 13. Depletion is less than for discrete white spots. Results of a hardness traverse across the white spot exhibited "hills and valleys," which is consistent with the dendritic morphology.

Small dendritic white spots (<2.5mm, 0.1 inches) such as the two shown in Figure 10 have been called "flecks," and can appear in clusters. Dendritic white spots usually - but not always - are "clean" (not associated with inclusions). Dendritic white spots are not found as often in alloys such as Waspaloy as they are in Alloy 718.

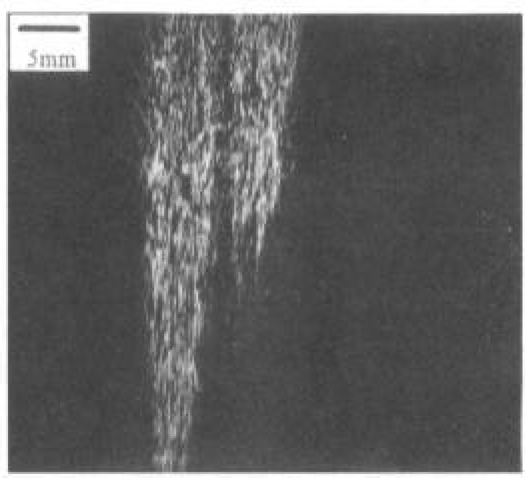


Figure 12. Cluster of Two Dendritic White Spots in an Alloy 718 Longitudinal Billet Slice. Canada Etch.

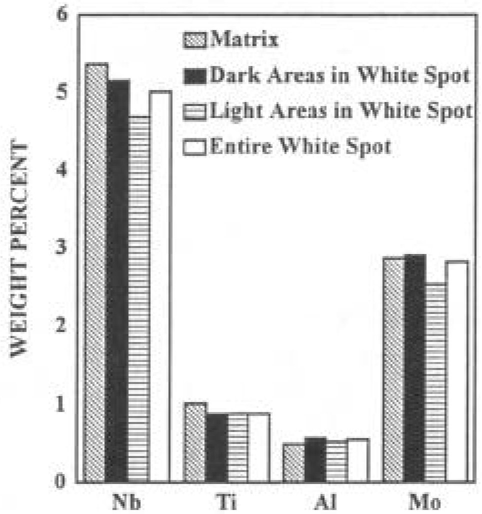


Figure 13. EDS Analysis of Depleted Elements in Different Areas of a Dendritic White Spot in an Alloy 718 Billet.

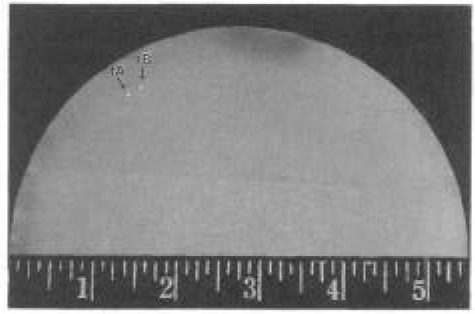


Figure 14. Two Solidification White Spots in an Alloy 718 Billet Slice. Canada Etch. Scale in Inches.

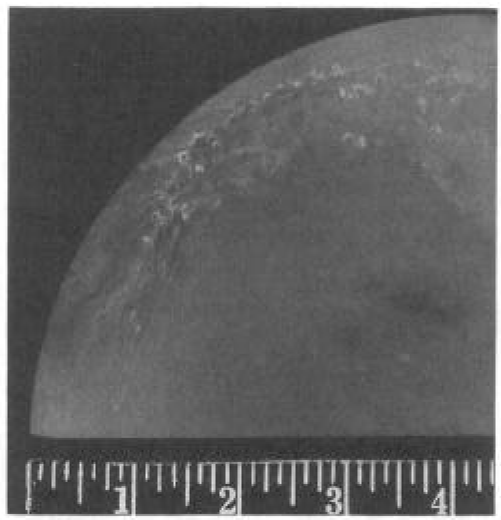


Figure 15. Numerous Solidification White Spots in an Alloy 718 Billet Slice from a Melting Experiment.

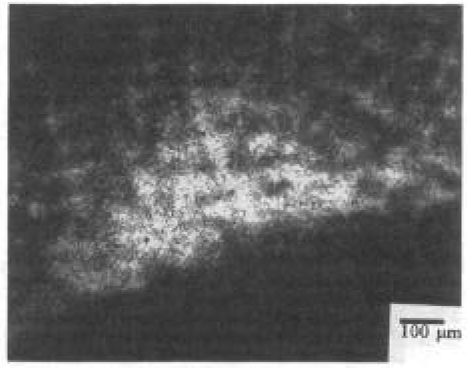
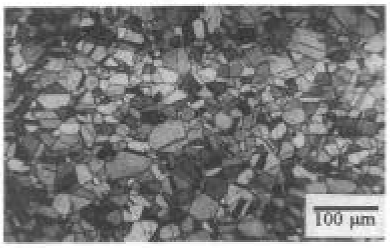


Figure 16. Solidification White Spot in an Alloy 718 Billet Slice. Note Diffuse Portions of Boundary. Canada Etch.

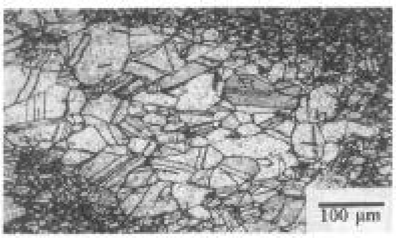
Solidification White Spots

It has only been in the past few years that solidification white spots have been recognized. They have been known by several names, including: "light-etching areas," "mini white spots," and "ring white spots." There was extensive discussion at the first White Spots Workshop before settling on "solidification white spot" as the preferred term. Typical solidification white spots in an Alloy 718 billet slice are shown in Figure 14. Note their linear appearance and their association with the ring pattern. Solidification white spots are always located between the surface and mid-radius, and are more prevalent at the extreme bottom of an ingot. Sometimes they have a hook-like appearance with the concave side of the hook almost always facing the center of the ingot, or a full-circle or donut shape. These various shapes are evident in Figure 15 which show an Alloy 718 billet slice from a melting experiment; the ingot portion represented by the billet slice was melted by a practice aimed at generating solidification white spots. Solidification white spots exhibit a diffuse rather than distinct boundary for portions of the interface between matrix and white spot. This characteristic is shown in Figure 16 for a solidification white spot in an Alloy 718 billet slice.

Thermomechanical processing history determines if there is a coarser grain structure in the white spot relative to the matrix. There is no grain size difference for the white spot in the Alloy 718 billet slice shown in Figure 17a, while there is a difference for the white spot in the Alloy 718 forging in Figure 17b.



(a)



(b)

Figure 17. The Effect of Thermomechanical Processing. Solidification White Spot in an Alloy 718 Billet Slice (a) Shows no Grain Growth, while that in an Alloy 718 Forging (b) Shows Grain Growth. Modified Kalling's Etch.

Chemical depletion in solidification white spots is very small. For example, the niobium level in an Alloy 718 solidification white spot frequently is within specification limits. This is illustrated in Figure 18, which shows EDS data for the white spot in Figure 17b.

Solidification white spots usually are not detected in forged parts unless the grain size is coarser than the matrix. Therefore, detection is dependent on forging practice. Since the delta solvus temperature for a white spot in Alloy 718 is slightly below that of the matrix, forging at temperatures in the interval between these two delta solvus temperatures promotes grain growth in the white spot but not in the matrix. On the other hand, forging below the delta solvus temperature of the white spot results in no grain size differentiation.

Detecting a solidification white spot in an as-cast ingot structure is difficult but possible. A solidification white spot in a slice from a large diameter ingot of Alloy 706 is shown in Figure 19.

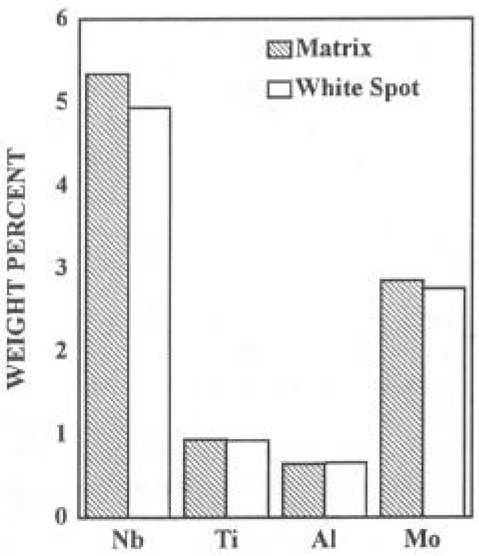


Figure 18. EDS Analysis of the Solidification White Spot in the Alloy 718 Forging Shown in figure 17b. Chemical Depletion is Very Small.

Figure 19. Solidification White Spot in Ingot Slice from Alloy 706 Ingot.

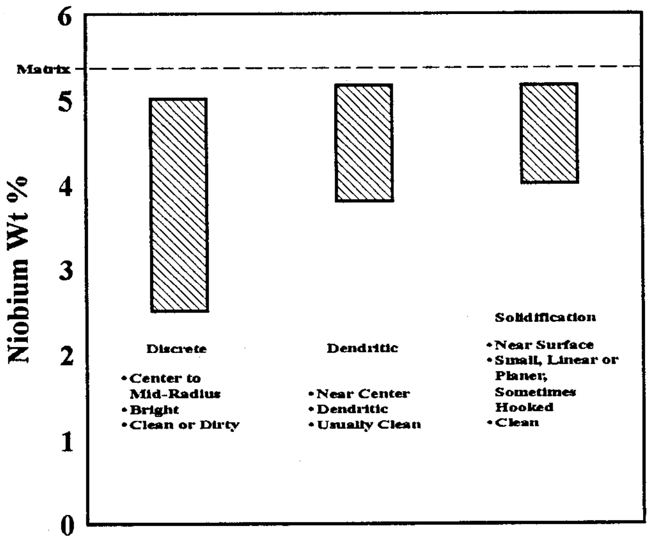


Figure 20. Summary of the Distinctive Features of the Three Types of White Spots. The Ranges of Niobium Levels that have been Reported for White Spots in Alloy 718 are Represented by Bars.

Summary: The classification of white spots is summarized in Figure 20.

Mechanisms of White Spot Formation

Extensive work has been done to understand the source of white spots and to minimize their occurrence in superalloys. It is widely accepted that white spots are associated with the inherent characteristics of the vacuum arc remelting (VAR) process. The mechanisms by which they form are believed to be different for each type. In general, discrete and dendritic white spots result from the "fall-in" of solid material into the molten pool in the VAR furnace. Solidification white spots, on the other hand, result from thermal perturbations that occur at the interface between the molten pool and solidifying ingot. Characteristics of white spots, such as their location in the ingot, composition, solidification structure, and cleanliness, are important clues to determining the mechanisms responsible for their formation.

Possible sources of discrete and dendritic (fall-in type) white spots during VAR processing are fall-in from the torus, crown, shelf, and from the pipe cavity of the electrode (Figure 21). High temperature gradients and molten metal convection are prevalent in the shelf and crown regions, resulting in planar solidification and draining of interdendritic fluid. The solute-depleted regions that form have a relatively high solidus temperature and a composition similar to the dendrite cores. The niobium gradient in the shelf region of a 510mm (20 inches) diameter Alloy 718 ingot is shown in Figure 22. The conditions for torus formation are similar.

Forming Discrete White Spots

The fall-in of shelf, crown, and torus material observed during vacuum arc remelting is postulated to be the source of discrete white spots. It occurs by mechanical detachment aided by pool turbulence. These solute-depleted regions can be entrapped in the solidifying VAR ingot because they have a higher melting temperature range than the base alloy and are denser than the liquid. Heat transfer models confirm that fall-in solids can fail to melt completely and can be subsequently entrapped by the advancing solidification front.[1] Compositional and microstructural analyses also support the fall-in hypothesis. For example, a comparison of Figures 5 and 22 shows that Alloy 718 white spots and shelf have similar niobium levels; also, the similarity between the shapes of Figures 4 and 22 are evident. It can be asserted that dirty white spots are likely to arise from sources such as shelf and crown where nitrides and oxides are observed to agglomerate.

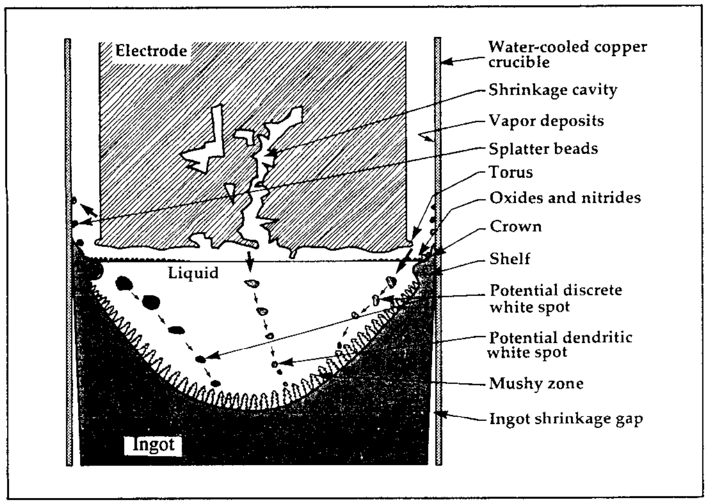


Figure 21. Schematic of the VAR Process Shows Potential Sources of Discrete and Dendritic White Spots.

Superalloy melters have found that increasing the melt rate results in deeper pools and reduced frequencies of discrete white spot formation. This is consistent with the fall-in mechanism because a deeper pool of molten metal provides more opportunity for remelting of solids that fall into it. However, the extent to which melt rates can be increased is limited by the onset of other types of segregation, such as "freckles" and severe dendritic patterns.

Forming Dendritic White Spots

Electrode fall-in is the mechanism most widely accepted for generating dendritic white spots. Fall-in is most likely to occur when electrodes contain solidification pipe, as shown in Figure 21. The pipe results in a change in electrode cross sectional area, a less stable arc, and erratic melting. These conditions can cause dendrite clusters to fall into the pool, resulting in dendritic white spots. In fact, the tendency to form dendritic white spots is apparently increased under most conditions that result in a constricted or unstable arc. Some confirmation of this theory is provided by the observation that dendritic white spots are not found in triple-melted Alloy 718, where the electrode is a solid electroslag remelted (ESR) ingot.

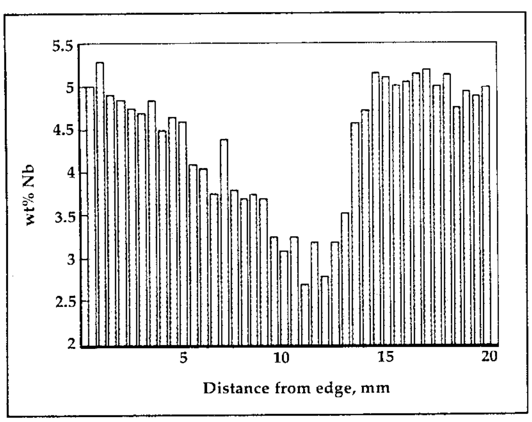


Figure 22. The Niobium Gradient in the Shelf Area of an Alloy 718 VAR Ingot.

Forming Solidification White Spots

Solidification white spots occur only in the surface to mid-radius region of a billet cross section, and form when a shallow molten pool is present during VAR processing. They are not believed to be the direct remnants of solids that fell into the pool. Instead, they are thought to result from a localized decrease or arrest in solidification rate. The slower rate allows coarsening of primary dendrite arms, which provides an opportunity for some interdendritic fluid to be swept away (see Figure 23[2]). What results is a region that is slightly leaner than the matrix in interdendritic solutes.

This mechanism explains the diffuse interface observed along portions of the solidification white spot shown in Figure 16. It also is consistent with the observation that solidification white spots frequently are associated with the "tree ring" patterns that consist of concentric rings highlighting pool profiles. It is widely accepted that these rings are the result of changes in solidification rate that cause variations in dendrite arm spacing. The observation that solidification white spots are clean also meshes with this mechanism. The prevalence of solidification white spots at the extreme bottom of the ingot can be explained by the shallow pool in this region: A shallow pool makes solidification more sensitive to thermal perturbations.

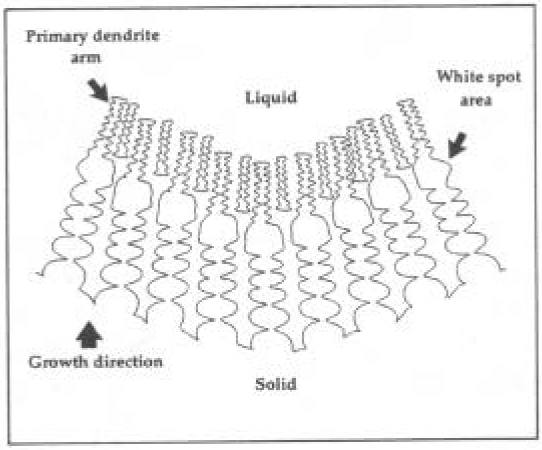


Figure 23. Schematic of the Mechanism that is Believed to Result in the Formation of Solidification White Spots. The Coarsened Dendrite Areas are Potential Sites.

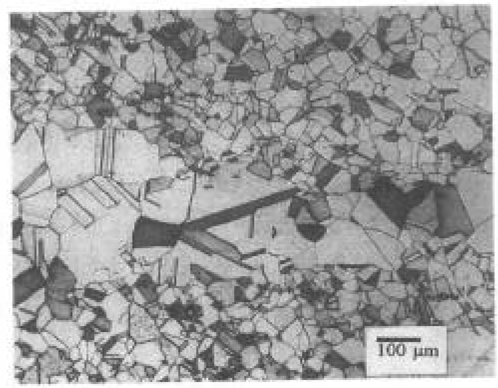


Figure 24. Coarse-Grained Area Generated in Alloy 718 by Heating a Billet for One Hour at 1010°C (1850°F).

TMP Can Produce Light-Etching Regions

Thermomechanical processing (TMP) can produce regions that etch lightly and have the appearance of white spots because of their coarse grain structures. However, these "pseudo white spots" are not solute-lean. An example is given in Figure 24, which shows the microstructure of an Alloy 718 billet slice heated at 1010°C (1850°F) for 1 hour. The grain structure was uniform prior to heating. The chemical composition differential between the coarse- and fine-grain regions is well within that observed when traversing any well-homogenized billet from a 510mm (20 inch) diameter ingot. Depending on the forging temperature and amount of reduction, coarse-grain regions such as that shown in Figure 24 may or may not remain coarse grained after forging into a part.

Alloy 718 is especially prone to the formation of this type of light-etching region because of the wide temperature interval over which delta phase goes into solution. A well-homogenized, fine-grain billet of standard-composition Alloy 718, produced from a 510mm (20 inches) diameter ingot, can begin to exhibit coarse-grain regions at temperatures around 995°C (1825°F), and a fully coarse-grain structure is not obtained until about 1030°C (1885°F). Therefore, forging within this temperature range can lead to light-etching regions if final reduction is not sufficient to cause complete recrystallization. Forging at temperatures below 995°C (1825°F) will not produce coarse-grain regions if the structure of the starting billet is fine and uniform. Thus, when forging superalloys, it is important for forgers to recognize that light-etching regions can be generated by thermomechanical processing at too high a temperature. Also, billet suppliers must provide material that is as homogeneous as possible.

How White Spots Affect Properties

The influence of white spots on tensile properties has been evaluated by transverse testing of Alloy 718 billet slices containing the defects.[3] Results of tests at room temperature are shown in Figure 25. The discrete and solidification white spot data are for individual tests, while the dendritic white spot results are the average of 10 tests. In each case, material adjacent to the white spot (the matrix) was tested for comparison. Also, all white spots were clean (without clusters of inclusions). The data for each property have been normalized to the dendritic white spot matrix value to simplify making comparisons.

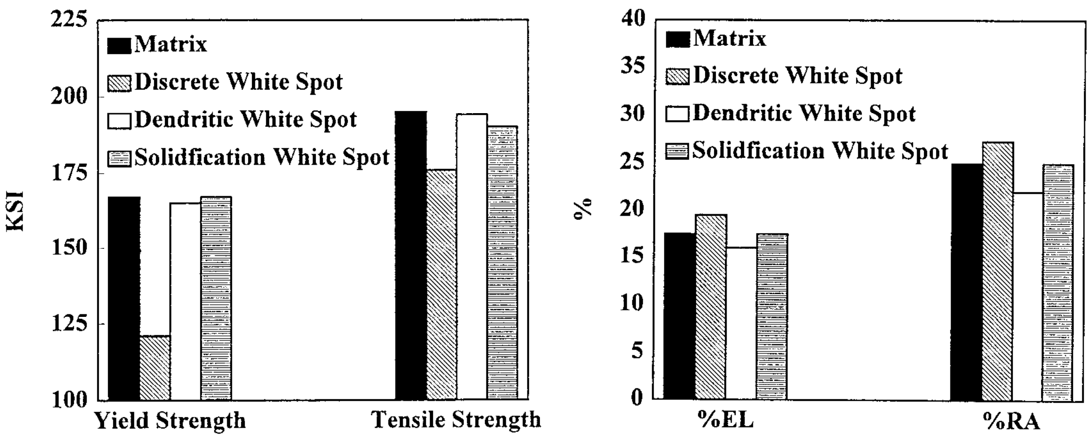


Figure 25. Effects of White Spots on Tensile Properties at Room Temperature of Specimens from Alloy 718 Billet Slices. Data Normalized to the Same Matrix Properties.

For material containing discrete and solidification white spots, strengths are lower but ductilities are higher relative to the matrix. The drop in strength for the solidification white spot is small because of the small difference in chemical composition. The strengths of specimens containing dendritic white spots are very close to those of the matrix and the ductilities are slightly lower. Results of tensile tests at elevated temperature 650°C (1200°F) show the same trends. Evaluations of billet slices having dirty discrete and dirty dendritic white spots show significant drops in ductility.

The possibility that the strength of a tensile test specimen having a white spot is equivalent to that of a uniform test specimen having the same hardener content (niobium, titanium, or aluminum for Alloy 718) as the white spot also has been studied.[4] The tensile strengths of two specimens having solidification white spots and a specimen having a discrete white spot agreed closely to a curve generated by multiple regression analysis of various Alloy 718 chemistries.

Fatigue data: Preliminary results from studies on the influence of white spots on low-cycle fatigue (LCF) properties have been obtained. Figure 26[5] shows results for cylindrical test specimens having a dirty discrete white spot and clean dendritic and solidification white spots. In each case, the matrix adjacent to the white spot was tested for comparison. No significant effect on LCF was found for the dendritic or solidification white spots. However, the LCF life of the specimen containing a dirty discrete white spot was significantly degraded.

Clean discrete white spots may have a slightly negative affect on LCF life as shown in Figure 27;[6] cycles to failure are slightly greater for the matrix than for the white spots. Grain size differences between the coarser white spots and the matrix were similar to the difference shown in Figure 3.

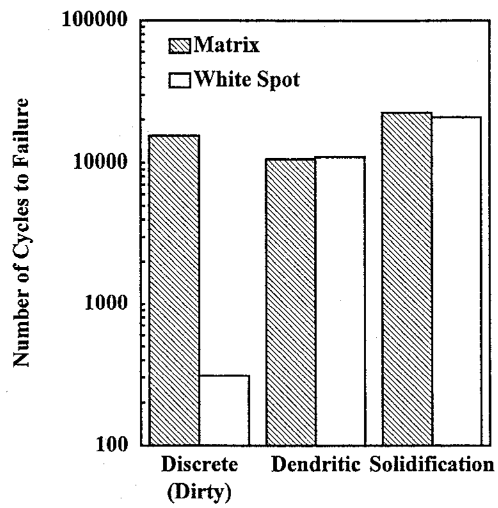


Figure 26. Influence of White Spots on the Low Cycle Fatigue (LCF) Life of Alloy 718 Billet Slices. Test Conditions: 315°C (600°F), A = 1.0, 0.4 Hz.

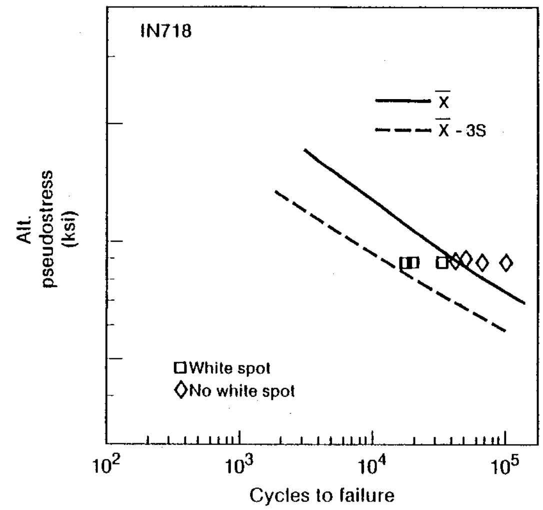


Figure 27. LCF Data for a Part with Clean Discrete White Spots in Fine Grain Alloy 718. Test Conditions: 371°C (700°F) for Curves and 400°C (750°F) for Data Points, A = 1.0, Strain Range = 0.67.

In Figure 27 , the solid line represents the mean curve for fine grain Alloy 718 at 371°C (700°F) and the dashed line represents minus three sigma. Specimens representing the data points were 5.13mm (0.202 inches) in diameter and were tested at 400°C (750°F); those with and without white spots were from adjacent material. Although cycles to failure were less with clean discrete white spots, all values were within three sigma. The decrease in life is attributed to the difference in grain size. Similar results were reported for clean discrete white spots that showed a drop in stress of about 27% under load control at 550°C (1022°F)[7]. The matrix grain size was ASTM 7 to 8, compared to the coarser white spot grain size of about 4. Two dirty discrete white spots fractured when loaded.

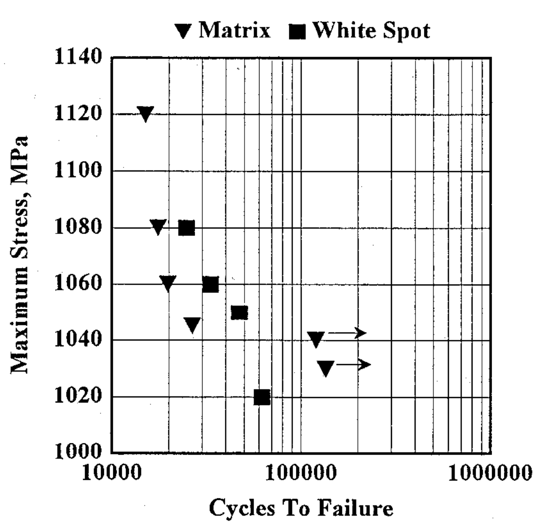


Figure 28. Influence of Solidification White Spots on LCF Life of Alloy 718 Forgings. Test Conditions: 400°C (750°F), Load Control, R Ratio = 0.

Additional preliminary data have been generated for solidification white spots, which are now the most prevalent type. In this study,[8] billets of Alloy 718 were produced via an experimental melting practice designed to create solidification white spots. The billets were forged into pancakes and macroetched. Flat LCF specimens were then prepared; some contained white spots, while others did not. Test results (Figure 28) indicate that solidification white spots are not deleterious to LCF life.

It is not surprising that dirty discrete white spots decrease LCF life, because inclusion clusters serve as sites for early initiation of cracks. Clean discrete white spots can decrease

LCF life but they are not disastrous. The decrease that has been observed is about the same magnitude as what would be predicted from the coarser grain size of the white spots. It also is not surprising that clean dendritic and solidification white spots appear to have no significant effect on LCF. In addition to not being associated with inclusions, these white spots have compositions very close to that of the matrix and continuity of microstructure prevails between white spot and matrix. Also, as indicated by tensile test data, the ductility of solidification white spots equals or exceeds that of the matrix. However, additional data are needed to further establish LCF properties for solidification white spots significantly coarser than the matrix.

Using Etchants to Detect White Spots

Surface-connected white spots can be detected using a variety of chemical and electrochemical etching methods. Four suitable etchants are listed in Table I.

Table I. Etchants for Detecting White Spots

Ferric chloride	$FeCl_3$ solution* HCl HNO_3	230 mL 750 mL 20 mL
Canada	HF HNO_3 H_2SO_4 H_2O	200 mL 100 mL 200 mL Make to 1 L
Dark etch	$FeCl_3$ HCl H_2O	489 g 37 mL Make to 1 L
Anodic	H_2SO4 H_2O	484 mL Make to 1 L

* 42% Fe solution: 550 g $FeCl_3$, make to 1 L with H_2O.

Development of the etching contrast needed to expose a white spot depends on composition and microstructure. The chemical composition difference between a white spot and the alloy matrix may not be sufficient to highlight an area. In most cases, however, white spots are readily exposed because of differences in the volume and distribution of precipitated phases. In Alloy 718, for instance, differences in the distribution and morphology of delta phase result in different etching characteristics. Canada etch, for example, will darken areas that are heavily precipitated, but leave solute-lean areas light. In Waspaloy, solute-lean areas that have fewer and finer gamma prime precipitates will also etch lighter. Since etching response is structurally as well as chemically dependent, the thermomechanical history of a part can influence the sensitivity of the selected etchant. For example, a white spot that is only slightly solute-lean may be difficult to detect if the alloy is in a fully solution heat treated condition.

More Mechanical Testing is Planned

Dirty discrete white spots cannot be tolerated because they can have a deleterious effect on mechanical properties, such as LCF life. Clean discrete white spots are not disastrous but they can decrease LCF life when their grain size is coarser than the matrix. It is believed that dendritic white spots can be eliminated by triple melting. They also are not commonly found in most alloy systems. Therefore, future efforts of the White Spots Committee will focus on solidification white spots. Emphasis will be placed on LCF testing and other mechanical property evaluations, such as the spin testing deemed necessary by gas-turbine engine builders. Close cooperation among materials suppliers, forgers, and engine builders will ensure that appropriate testing parameters and specimens are used. Also planned are metallographic evaluations of fracture test specimens to identify initiation sites and characterize white spots. Emphasis will be placed on documenting the grain size of the white spots relative to their matrix. These programs will be organized by the Mechanical Property Subcommittee.

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4. Reported at April, 1991 White Spots Workshop by L. W. Lherbier representing Cytemp Specialty Steel.

5. Reported at May, 1992 White Spots Workshop by S. J. Choe of Textron Lycoming.

6. Reported at April, 1991 White Spots Workshop by J. F. Barker representing General Electric.

7. Reported at May, 1992 White Spots Workshop by S. Besse of SNECMA.

8. Provided by P. Spink of Rolls Royce to the Mechanical Property Subcommittee of the White Spots Committee.