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The effect of VAR process parameters on white spot formation in INCONEL[1] 718

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A 20 inch diameter VAR ingot of Inconel 718 was produced at Special Metals Wiggin Limited (Hereford) under conditions of abnormally long arc gap. It was sectioned and analyzed in the as-cast state. White spots were found, along with unusual macrostructural features at the ingot surface. The experimental results are presented and discussed in relation to possible formation mechanisms, and it is shown that the level of niobium found in these white spots was too low to be explained by conventional mechanisms of formation. © *2004 Kluwer Academic Publishers*

1. Introduction
Vacuum Arc Remelting (VAR) is an important secondary re-melting process that is widely used for modern turbine disc alloys to:

- refine the structure,
- improve the cleanness, and
- reduce defects.

The last is of great importance as the yield strength of turbine disc alloys is increasingly sensitive to the presence of defects. Although improved control of melting processes has reduced the incidence of many melt-related defects (e.g., freckles, solute-lean defects or white spots) such defects are still occasionally found in VAR material. As a result, extensive work has been done to understand the sources and formation mechanisms of white spots, to minimise their occurrence in superalloys.

White spots in alloy 718 are recognizable as light etching spots on a macro-etched surface, and are lower in alloying elements such as titanium and niobium. White spots have been generally split into three categories: discrete white spots, believed to be associated with undissolved material which has fallen in from the shelf, crown or torus regions; dendritic white spots, usually associated with residual unmelted dendrites from the consumable electrode; and solidification white spots, believed to be caused by local perturbations in the solidification conditions [1, 2]. This paper concentrates on the effect of the length of the arc during VAR—the ‘arc gap’—on white spot formation.

A number of white spots were found in a production scale as-cast ingot of INCONEL 718 that had been produced with an abnormally long arc gap following an equipment problem. The relationships will be discussed between their formation mechanism, the process operating conditions, and the physical processes occurring during VAR. The ultimate goal is to gain a better understanding of the links between process parameters and final ingot structure. It is emphasized that in this investigation the white spots were studied in the as-cast state, which is unusual, because they are normally detected and analysed following forging.

2. Experimental method
An aborted production scale (0.5 m dia) ingot from Special Metals Wiggin Ltd., Hereford, was sectioned in order to gather data concerning the effects of the process conditions. This ingot had been made with a progressively increasing arc gap due to an electrode drive problem, but it was decided to continue the melt for experimental investigation. The actual process parameters and estimates of arc gap are shown graphically in Fig. 1. After the start-up stage, the melting current was held at 6 kA, and the arc gap varied from 10 to 210 mm. When the arc gap reached 210 mm, it was rapidly decreased to 1 mm; then it increased until it reached 150 mm. The ingot was sectioned transversely into sections about 30 cm tall. Each of these sections was cut longitudinally along the nominal ingot centreline to yield diametral slices. Each of these slices was cut in two to give two half slices from the left and right sides of the melt pool. The left hand slices were macro-etched using acidic ferric chloride to reveal the grain structure. The right hand slices were electrolytically etched in 50% HCl, followed by an electrolytic stain in 4% H_2SO_4 to reveal the segregation features. Three such etched sections are presented to scale with the process data in Fig. 1. Samples for optical microscopy and SEM analysis were cut from these longitudinal slices and

[1]INCONEL is a trade mark of Special Metals Corporation.

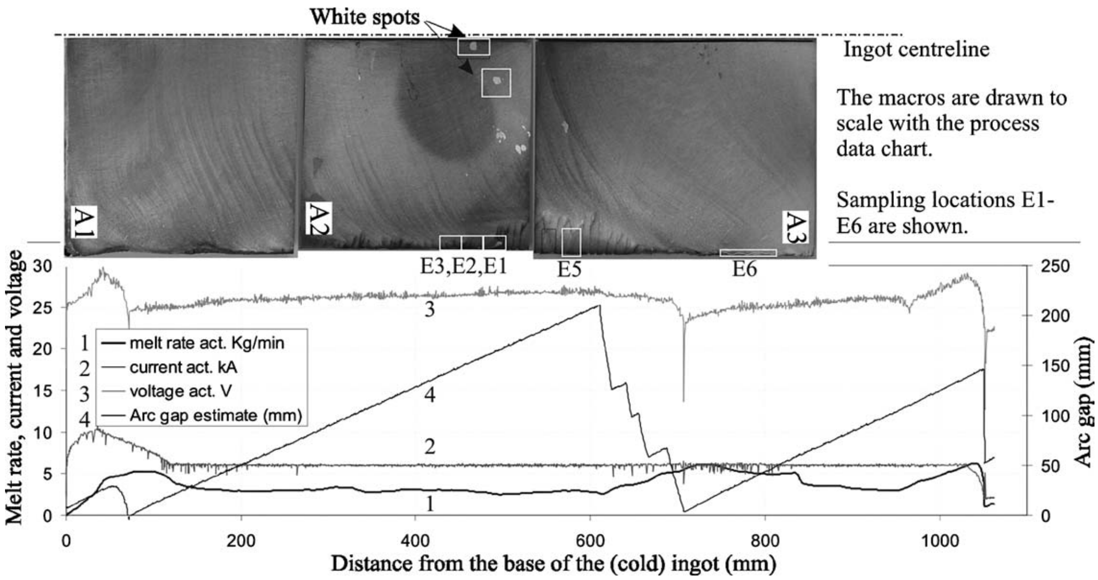

Figure 1 VAR process data and the resulting ingot macrostructure. The scale is indicated by the *x* axis of the process data chart.

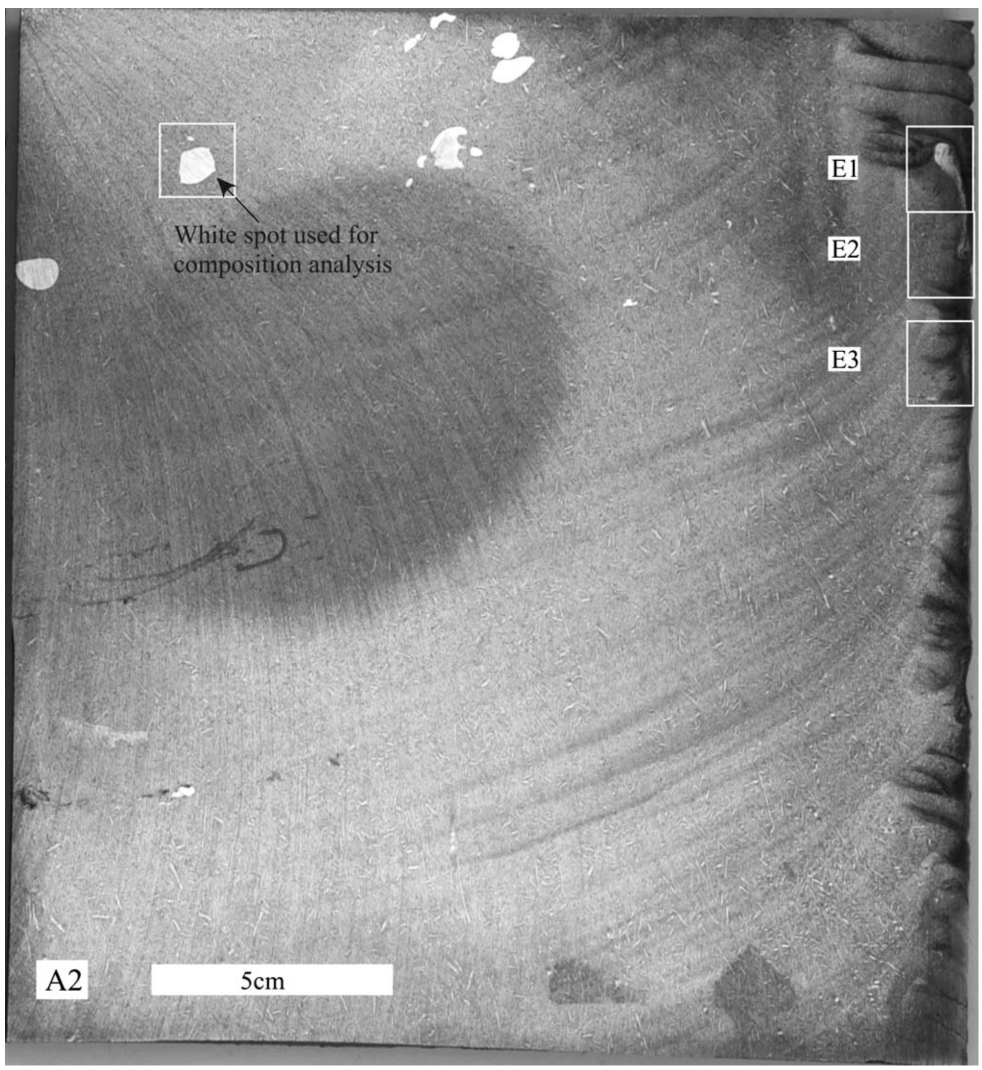

Figure 2 Slice A2, showing white spots. A close up of the region containing sample locations E1, E2 and E3 is shown in Fig. 5.

diamond polished to a 1 μm finish. Samples for optical and electron microscopy were etched using Kallings etchant. The compositional analysis (on as-polished samples) was conducted on a Jeol-6300 scanning Electron Microscope (SEM) equipped with an EDS facility. The operating voltage was 20 kV and the current of the condensing lens was 0.3 nA. The live sampling time for each analysis was 100 s. Some samples were also analysed on an Oxford 7430 SEM with a WDS facility.

3. Experimental results

In this work, samples were metallurgically examined from three regions of the as-cast ingot: (1) from a region that contained discrete white spots; (2) from areas at the edge of the ingot which could be sources of white spot materials (areas E1, 2, 3, 5 and 6 of Fig. 1); (3) from the ingot 'crown'. Results from these three regions are presented below.

3.1. White spots

From Fig. 1, it can be seen that there are a number of white spots in the mid-radial to central locations of slice A2 and one white spot in slice A3. In order to see these white spots more clearly, slice A2 is shown in higher magnification in Fig. 2. The white spots appear bright and have distinct boundaries, with irregular shapes and different sizes.

One white spot sample was cut from the location shown in Fig. 2. The polished sample was analysed by EDS (using at least 5 area measurements of 1 mm^2 each) and the results are shown in Fig. 3. The sample was also analysed using WDS to check these measurements and no significant differences were found. It can be seen that the white spot was depleted in niobium, titanium and molybdenum, although the aluminium level appears to be similar to that of the matrix. The niobium content was very low in this white spot (<1 wt%), which is quite different from previous published data (typically 2–3 wt% [1, 3]). [In order to verify the unexpected composition measurements, other white spots from this ingot were also sectioned and analysed using both EDS and WDS, but similar low levels of Nb (<1 wt%) were found]. The sample was then etched and used for SEM observation, and a SEM micrograph is shown in Fig. 4.

From Fig. 4 it can be seen that the white spot consists of non-segregrated, equiaxed grains, whereas, the matrix has a clear, segregated dendritic structure.

3.2. Analysis of surface regions

Discrete white spots are generally thought to result from pieces of shelf, crown and torus falling in from close to the edge of the ingot. Therefore, possible sources

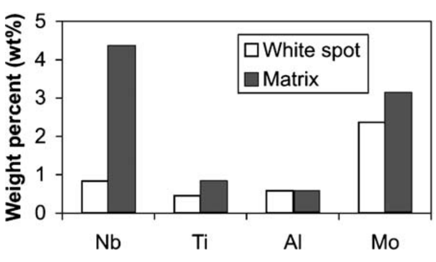

Figure 3 Results of compositional analysis of the white spot shown in Fig. 2

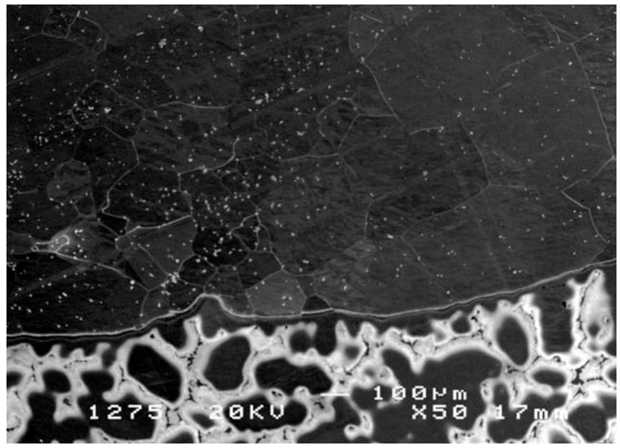

Figure 4 SEM image of the white spot (upper) and the surrounding matrix (lower).

of white spots were first sought on the edge area of the ingot. Several samples were cut from the edge of slices A2 and A3 and analysed (using 5, 1 mm^2 area measurements as described previously) and the results are presented in Table I. The sample locations are shown in Figs 5 and 7. From Table I, the niobium content was found to be in the range of 4.64–5.52 wt% in the 'white' and 'black' regions of all three samples. The 'grey' region in E2 had a high Nb content ($\sim$12 wt%).

In order to investigate variations in the Nb, Mo, Al, Ti content of the E1 'white' region and the surrounding area, a series of spot analyses (point measurements with a stationary electron probe) were made along a line at the location shown in Fig. 5, with a step size of 2 mm. The results are shown in Fig. 6. Although some variations are visible, especially in the niobium content, these may well be due to sampling interdendritic regions. It can be seen that the mean niobium value is close to that of the matrix.

Sample E5 was cut from the location shown in Fig. 7, and the 'triangle' region analysed by EDS. The composition is shown in Table II. This region was found to be rich in carbide and Laves phases. Spot analysis was also carried out along the arrow shown in sample E5, and measured variations in Nb content are shown in Fig. 8. It can be seen that the Nb content was between 3.5 and 7.8% at most points. No areas of low Nb content (<1 wt%) were observed.

The search for the source of white spots was extended to a second area, again at the edge of the ingot, marked as 'E6' in Fig. 7. Sample E6 contained a thin, wavy light–etching region with what appeared to be normal matrix on either side. The light feature and the adjacent matrix were analysed (using area measurements) and the results are presented in Table III.

Somewhat surprisingly there appeared to be only a small difference in composition between the light feature and its surrounding matrix.

3.3. Analysis of crown material

Samples of material from the crown from the top of a typical ingot were analysed using Inductively Coupled Plasma Optical Emission Spectroscopy. A

TABLE I E1, E2, E3 composition (wt%) with standard deviation

	Element	Ti	Cr	Fe	Ni	Al	Nb	Mo	Si
E1 White region	Mean	1.06	19.23	17.53	52.98	0.7	5.15	3.23	0.22
	S.D.	0.25	0.34	0.38	0.48	0.12	0.85	0.17	0.07
E2 Grey region	Mean	1.24	16.36	13.99	51.93	0.54	12.03	3.66	0.25
	S.D.	0.19	0.63	0.54	1.68	0.21	2.65	0.39	0.09
E2 Black region	Mean	1.13	19.57	17.56	52.91	0.63	4.64	3.37	0.19
	S.D.	0.11	0.43	0.26	0.40	0.04	0.30	0.15	0.04
E2 White region	Mean	0.98	19.63	17.04	52.71	0.74	5.52	3.14	0.23
	S.D.	0.11	0.18	0.27	0.67	0.09	0.33	0.23	0.11
E3 White region	Mean	1.29	19.54	17.58	52.58	0.75	4.65	3.35	0.26
	S.D.	0.50	0.11	0.20	0.55	0.17	0.04	0.25	0.10

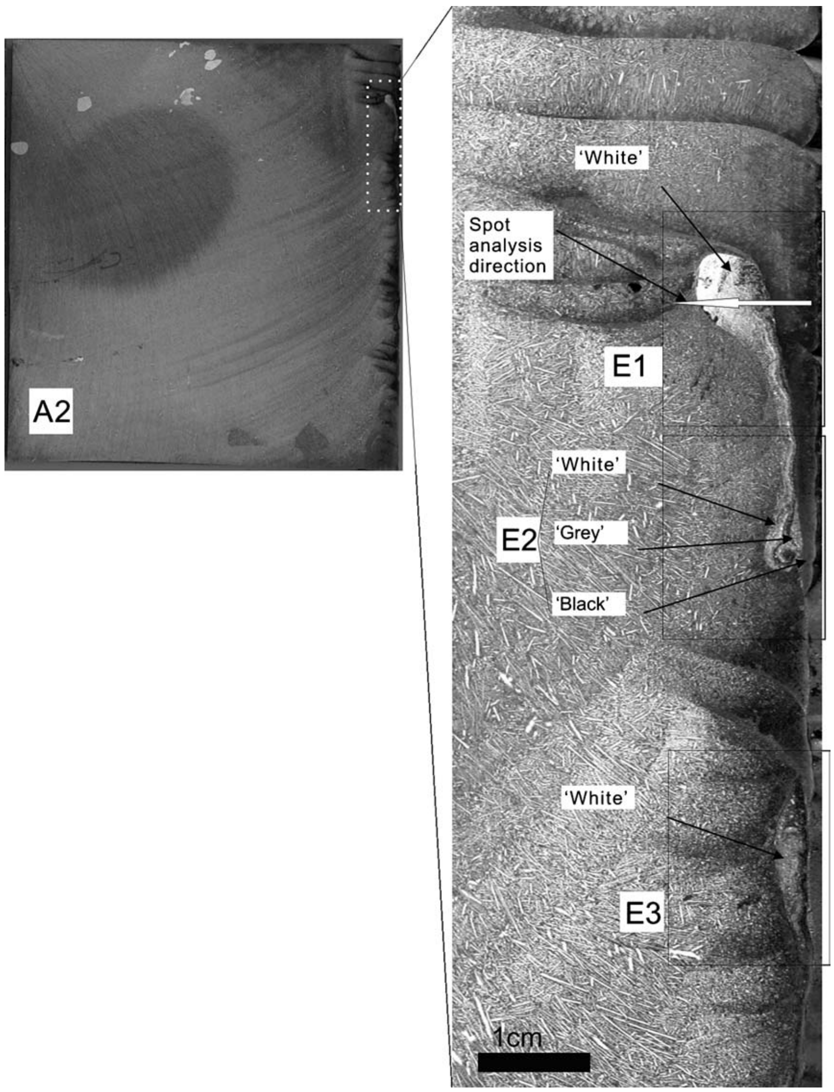

Figure 5 Enlarged image of a region from the edge of slice A2 (Fig. 2) containing samples used for analysis.

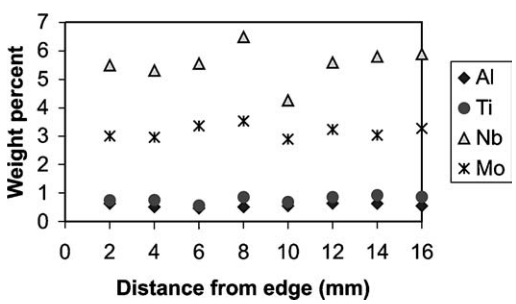

Figure 6 The results of spot measurements along the line indicated in area E1.

range of levels of aluminium were measured in the different samples, but the minimum observed was 2.3 wt%.

4. Discussion

The major experimental result from this investigation is that the observed white spots were unusually low in niobium content (less than 1 wt% compared with the normal 2–3 wt%). Consequently, it is interesting to speculate on possible mechanisms of formation.

TABLE II Composition of triangle region in sample E5

	Element	Ti	Cr	Fe	Ni	Al	Nb	Mo	Si
E5	Mean	1.25	17.76	14.51	51.37	0.55	10.57	3.74	0.25
	S.D.	0.16	0.29	0.33	0.79	0.07	0.73	0.32	0.09

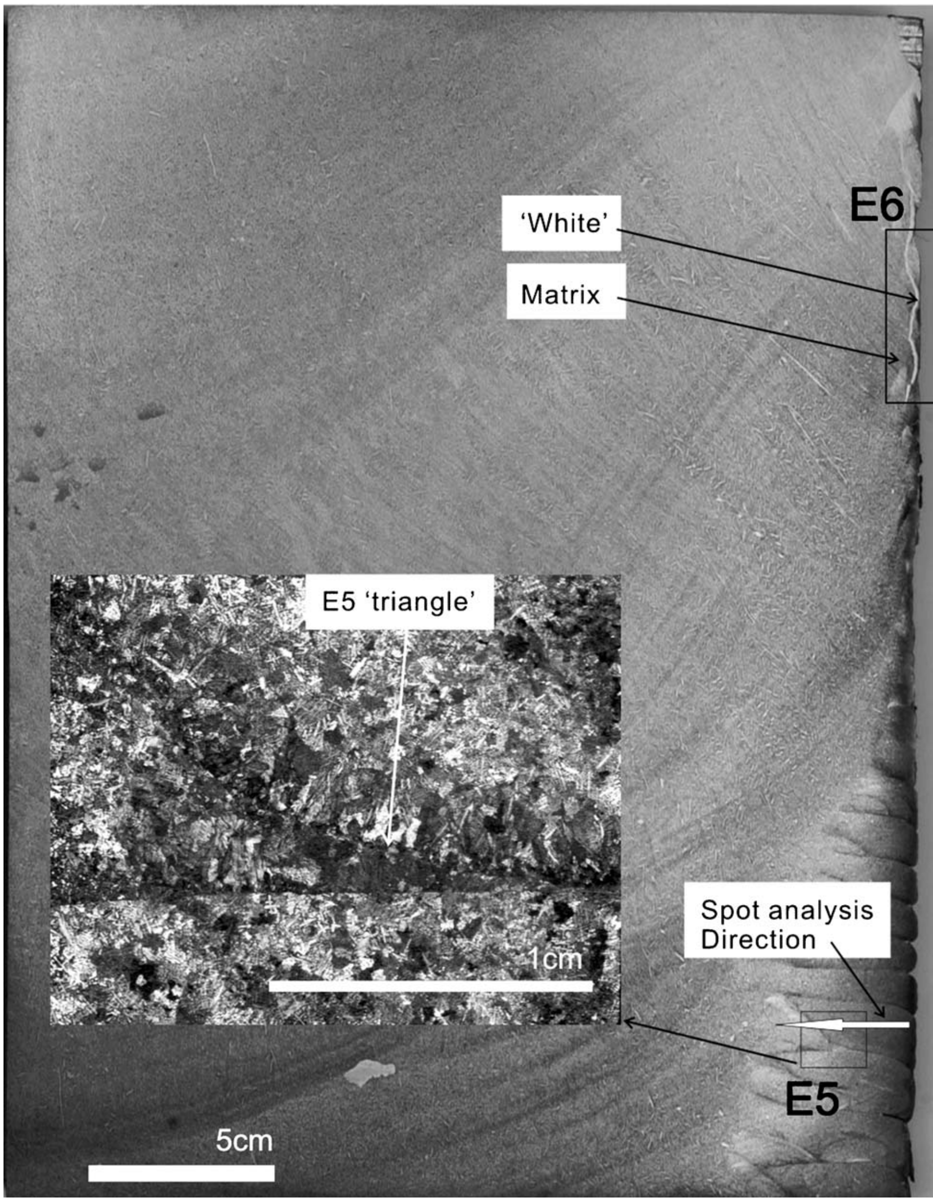

Figure 7 Samples E5 and E6 cut from Slice A3.

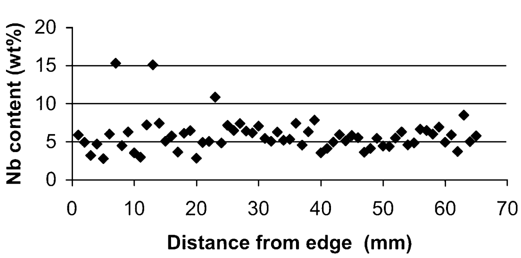

Figure 8 Variation in measured Nb content along a line of spot analyses in the shelf area (E5).

Historically, people have suggested that discrete white spots are formed by the detachment and transport of material from the shelf, crown or torus regions. Low niobium concentrations are thought to arise in the shelf and torus from solidification-based mechanisms. From the pseudo-equilibrium phase diagram for 718 (Nb/alloy remainder) it can be seen that solid in contact with liquid of 5.2–5.5 wt% niobium should not contain less than 2–4 wt% Nb. This was the composition range observed by Yu *et al.* [3] in both white spots and the shelf area, suggesting that the shelf is a source of white spots. Samuelesson *et al.* [4] analysed torus material and found a niobium content of 3.9 wt%, which supports the theory that torus fall-in can cause white spots. However, the extremely low levels of Nb (<1 wt%) observed in this experiment are not consistent with these mechanisms.

Evaporation and condensation of material on the crucible wall could be a possible source of low niobium

TABLE III Composition of wavy, light-etching region and surrounding matrix (wt%) in sample E6

	Element	Ti	Cr	Fe	Ni	Al	Nb	Mo	Si
E6 wavy region	Mean	0.59	19.17	17.34	53.52	0.54	5.67	3.01	0.18
	S.D.	0.04	0.30	0.01	0.18	0.01	0.28	0.15	0.05
Matrix	Mean	0.83	19.51	17.34	53.46	0.54	4.89	3.23	0.19
	S.D.	0.31	0.17	0.11	0.49	0.06	0.13	0.05	0.01

material. However, analysis of crown material in the present study showed aluminium levels in excess of 2 wt% (compared with approximately 0.5 wt% nominal in the matrix), which could be expected due to aluminium's high vapour pressure. In marked contrast, the measured aluminium content of the white spot analysed in this work was close to 0.5 wt%.

It is very likely that the long arc gap is implicated in the formation of the white spots observed here. However, the precise mechanism for this has not yet been identified. The long arc gap is probably responsible for the lapped macrostructural features observed at the edge of the ingot in some areas due to its effect on heat transfer. When the arc gap is short the arc behaves as a macrouniform diffuse source of heat that can only lose energy by radiation into a small exposed area of the crucible wall, and hence most of its power is transferred to the electrode and ingot. Therefore very little shelf is formed at the periphery of the ingot. However when the arc gap is large, the arc tends to become more constricted towards the ingot centre. Furthermore, as a large area of crucible wall is now exposed, a large amount of the arc's energy can be lost by radiation, further cooling the edge of the melt pool. Hence an unusual vacuum arc and temperature environment is created under the rare condition of a long arc gap. It seems that this condition promotes the formation of white spots with unusually low niobium contents, but the exact mechanisms are not clear as yet.

5. Conclusions

White spots with abnormally low niobium contents have been observed in an Inconel 718 VAR ingot produced under conditions of abnormally long arc gap. This observation has been discussed in the light of published mechanisms of white spot formation that involve material falling into the melt from the shelf, crown and torus regions. It has not been possible to use any of these published mechanisms to account for the abnormally low niobium contents observed. However the fact that these white spots are likely to have formed through a process different to those described so far may shed light on new aspects of VAR melting.

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References

1. L. A. JACKMAN, G. E. MAURER and S. WIDGE, *Adv. Mater. Proc.* **5** (1993) 18.
2. B. K. DAMKROGER, J. B. KELLEY, M. E. SCHLIENGER, J. A. VAN DEN AVYLE, R. L. WILLIAMSON and F. J. ZANNER, in "Superalloys 718, 625 and Various Derivatives," edited by E. A. Loria (The Minerals, Metals & Materials Society, 1994) p. 125.
3. K. O. YU, J. A. DOMINGUE, G. E. MAURER and H. D. FLANDERS, *JOM* **1** (1986) 46.
4. E. SAMUELESSON, J. A. DOMINGUE and G. E. MAURER, *ibid.* **8** (1990) 27.

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