THE INFLUENCE OF VIM CRUCIBLE COMPOSITION, VACUUM ARC REMELTING, AND ELECTROSLAG REMELTING ON THE NON-METALLIC INCLUSION CONTENT OF MERL 76

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A program was conducted to determine the effect of selected melt variables on the oxide inclusion content of MERL 76 ingot material. Melt techniques investigated included vacuum induction (VIM), vacuum arc remelting (VAR, and electroslag remelting (ESR). Results of the VIM study revealed that magnesia crucibles produced ingot material having the lowest oxide content whereas zirconia crucibles resulted in the highest oxide content. Vacuum arc remelting was shown to have little or no effect on reducing the oxide content of the starting ingot, whereas ESR was shown to significantly reduce oxide content. A technique for quantitatively assessing oxide content of an ingot was also developed and demonstrated.

INTRODUCTION

Studies to date have shown that the minimum low cycle fatigue (LCF) life of P/M nickel-base superalloy materials is set by the presence of small, non-metallic oxide inclusions in the material[1]. A reduction in the quantity and size of the inclusions would substantially improve the durability characteristics of P/M components, particularly gas turbine engine disks. The major sources for inclusions in P/M superalloy materials are 1) the ingot material from which the powder is produced and 2) the powder making process. Ingot material is usually produced by vacuum induction melting (VIM) in ceramic lined crucibles. Vacuum induction melting results in substantial refinement of the input material including deoxidation as a result of exposure of the melt to vacuum and melt reactions between oxygen and carbon. However, numerous sources for oxide contamination can be identified during VIM. These include complex melt reactions between the elemental additions and dissolved oxygen in the melt with the more thermodynamically

stable oxide specie predominating[2]. Reactions between the melt and crucible also acts as a source for inclusions as well as erosion and spallation of the crucible lining and tudish[2]. Interactions between the melt and crucible would be expected to be a sensitive function of the specific composition and integrity of the crucible material used. The initial phase of this study was, therefore, directed toward evaluating the effect of crucible variations on the inclusion content of MERL 76 VIM ingots. MERL 76 is an advanced hafnium bearing, P/M nickel-base superalloy.

Vacuum arc remelting (VAR) and to a lesser extent electroslag remelting (ESR) are frequently employed as secondary melt practices for nickel-base superalloys. Although the effects on ingot structure and chemistry of these melt practices are well known, little quantitative information with respect to effects on inclusion level in superalloys can be found[3]. The second phase of this study evaluated the effect of VAR and ESR on the inclusion content of MERL 76.

At the onset of this study, it became readily apparent that a technique for quantitatively assessing inclusion content in superalloy ingot was not available. Metallographic sectioning was not considered to be a viable evaluation technique due to the extremely small volume of material analyzed and the inherent error associated with the evaluation of a three dimensional particle on a two dimensional plane. Therefore, a final objective of this study was the development of a method by which the inclusion content of an ingot could be determined on a semi-quantitative basis.

EXPERIMENTAL PROCEDURE

Vacuum Induction Melting

Ten 136 kilogram (300 pound) heats of MERL 76 (see Table 1 for MERL 76 chemistry) were produced by vacuum induction

Table 1. Nominal MERL 76 Alloy Composition

C	Ni	Cr	Mo	Cb	Al	Ti	Hf	B	Zr
0.02	Bal	12.40	3.10	1.65	5.00	4.30	0.75	0.02	0.06

melting with the major variable being refractory composition. The refractories evaluated consisted predominantly of alumina (Al_2O_3), magnesia (MgO), or zirconia (ZrO_2) and included high purity preform crucibles as well as rammed or bricked crucibles

(see Table 2). All heats were produced from common lots of raw material and identical melt procedures were followed for each heat. A nickel wash heat was initially melted to condition the crucible. One additional heat (#4) was produced of the MERL 76 alloy less the most reactive elements aluminum, hafnium, titanium and zirconium. For comparison purposes a baseline heat (#6) was produced in a magnesia crucible which had previously been used to produce seven heats of various nickel-base superalloys. All heats were poured through a high alumina (94% Al_2O_3, 5.5% CaO) castable tundish into steel ingot molds having a diameter of 7 cm (2.75 in). Each heat produced five 24.5 kilogram (60 pound) ingots with the exception of the zirconia brick heat which was melted as two separate 68 kilogram (150 pound) pours each yielding three ingots having a weight of 23 kilograms (50 pounds). The as-cast ingot surface was maintained on all ingots.

VIM Ingot Cleanliness Evaluation

The cleanliness evaluation technique developed for use in this program consists of the following steps: 1) systematic sampling of each heat to provide a 3 pound sample for evaluation, 2) electron beam skull melting of the sample utilizing the principal of floatation to concentrate the oxides on the remelted ingot surface, 3) electrochemical extraction of the remelted ingot surface followed by digestion and filtration, 4) gravimetric measurement of oxide content. The three pound sample from each heat was made by removing a full cross sectional ingot slice from the top, middle, and bottom of the first, middle, and last ingot poured from each heat. Each slice was approximately 6 mm (0.25 in) thick and the cut was made with a non-oxide (SiC) abrasive wheel to prevent oxide contamination.

Electron beam remelting of nine ingot slices (collectively) from each heat was conducted in a hemispherical water cooled copper crucible having a diameter of 10 cm (4 in). A vacuum of 10^{-5} Torr is maintained while the sample is melted using a 60 KW electron gun. The sample is held molten for approximately one minute followed by a gradual powerdown to maintain a quiescent melt surface to promote floatation of the oxides. In order to insure that the entire ingot sample is melted, the ingot is inverted in the copper crucible and melted a second time using the same procedure. Examination of the surface of the EB remelted ingots revealed an oxide raft the area of which was measured to provide a qualitative indication of ingot inclusion content. Figure 1 shows the EB ingot from Heat #2.

Following EB remelting, the ingot is prepared for electrolytic extraction. A silicon rubber gasket is cast around the

Table 2. Composition of VIM Crucibles Used For The Superalloy Heats Evaluated

Heat No.	Crucible	Type	Composition Wt.%					
			Al_2O_3	MgO	ZrO_2	SiO_2	CaO	Fe_2O_3
1	Al_2O_3	Prefired	99.55	-	-	0.07	-	0.09
2	Al_2O_3	Rammed	94.00	4.80	-	0.50	0.18	0.08
3	MgO	Prefired	1.80	97.30	-	0.60	0.10	0.10
4[a]	MgO	Rammed	6.50	92.00	-	1.30	-	-
5	Same as Heat #4							
6[b]	MgO	Rammed	20.00	74.00	-	3.50	0.80	0.60
7	Spinel	Rammed	70.60	28.20	-	0.30	0.35	0.05
8	ZrO_2	Prefired	0.30	3.20	94.70	1.00	0.40	0.20
9	ZrO_2	Brick	0.30	3.20	94.70	1.00	0.40	0.20
10	Same as Heat #9							

a) MERL 76 less reactive elements

b) Baseline used MgO crucible

edge of the ingot which acts to electrically insulate the ingot (anode) from the cell (cathode) as well as forming a cavity for the electrolyte. The ingot is placed in an inverted position on the cathode which is made from commercially pure titanium. Two holes in the cathode provide inlet and outlet ports for the electrolyte which is composed of 20% HCl and 5% HNO_3. A current of 60 amperes is applied to the extraction cell and the electrolyte is alternately pumped by vacuum from one flask, through the cell and into a second flask maintaining the electolyte temperature at 50°C. This process continues until approximately 50 gms are removed uniformly from the surface of the ingot (approximately 0.2 cm). A second 50 gm sample is removed in a similar manner and the resultant electrolytes are combined, and the residue allowed to settle overnight. The electrolyte is decanted and the residue treated with selected acids in several steps to remove the majority of the non-oxide contaminants. The residue which consists predominantly of oxides and some carbides is collected on an 8 micron unipore filter for weighing and analysis. This residue, from the top 100 gm sample of the EB remelted ingot is identified as Layer "A". A second 100 gm sample is prepared in a similar manner and is identified as Layer "B". While the overwhelming majority of the oxides are usually found in the "A" layer, the "B" layer is taken to provide a check on the reproducibility of the extraction and digestion processes. The weight of the residue for each sample is determined by substracting the weight of the filter before and after the oxide is filtered. A summary of the gravimetric results for the VIM heats evaluated are presented in Table 3 along with measurements of the area of the floating oxide raft. Figure 2 shows the extraction apparatus.

VAR and ESR Melting and Cleanliness Evaluation

Based on the results of the VIM crucible study, MgO rammed was selected as the best practice. One heat was produced using identical melt procedures to those used previously. Sampling technique for establishing cleanliness of the VIM heat was similar to that previously used except that slices were only taken from the top and bottom of the VIM ingot.

Vacuum arc remelted ingots were produced from the VIM ingots using three different melt rates, namely 0.65 Kg/min (1.4 lbs/min), 1.1 Kg/min (2.4 lbs/min) and 1.9 Kg/min (4.2 lbs/min). One ingot having a diameter of 10 cm (4 inches) and a weight of approximately 2.25 Kg (60 pounds) was produced for each melt rate. In addition, one 10 cm (4 inch) diameter ingot was produced by electroslag remelting using a slag chemistry of 70% CaF_2 - 15% CaO - 15% Al_2O_3 and a melt rate of 0.59 Kg/min (1.3 lbs/min).

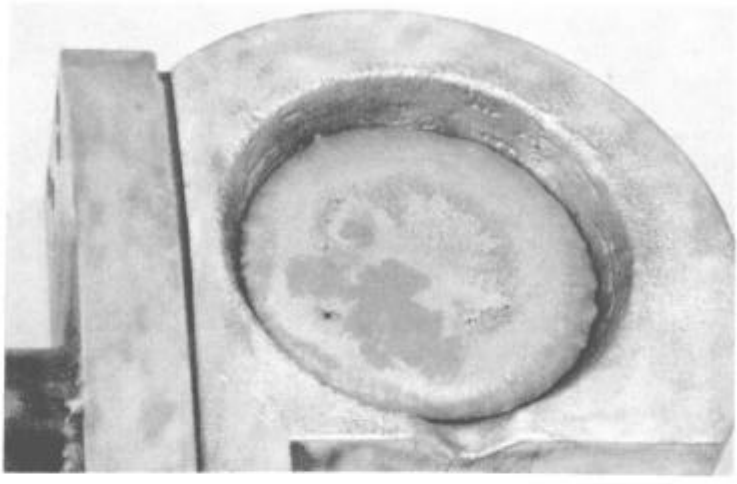

Figure 1. EB Remelted Ingot of the Al_2O_3 Rammed Crucible Heat (#2) Showing the Dark Oxide Raft Concentrated on the Ingot Surface.

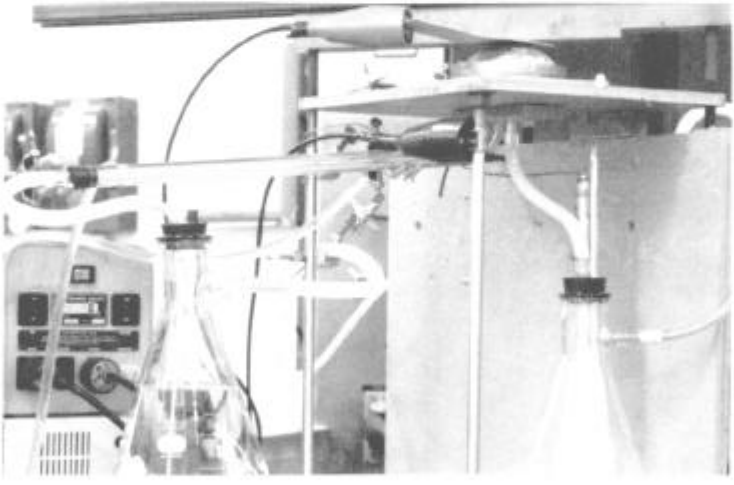

Figure 2. Electrolytic Apparatus Used to Extract the Oxides From the EB Remelted Ingot Surface. Note the EB Ingot on the Top of the Cell.

Table 3. Extraction Results From VIM Crucible Study

Heat No.	Crucible Type	Residue Weight (mg) "A" Layer	Residue Weight (mg) "B" Layer	Approx. PPM Oxide[b]	Area Of Raft[c]
1	Al_2O_3 Prefired	2.6	0.1	2	2
2	Al_2O_3 Rammed	9.7	0.6	8	4
3	MgO Prefired	8.9	0.2	6	4
4[a]	MgO Rammed	0.7	0.3	<1	0
5	Same as #4	5.8	0.3	4	2
6	MgO Rammed	2.6	0.2	2	1
7	Spinel Rammed	8.6	0.1	6	7
8	ZrO_2 Prefired	13.4	0.4	8	4
9	ZrO_2 Brick	11.8	0.2	8	8
10	Same as #9	14.6	0.3	9	10

(a) MERL 76 less reactive elements

(b) Weight of "A" layer divided by sample weight

(c) Normalized to heat #10 = 10

The VAR and ESR ingots were samples for cleanliness evaluation by taking full cross-sectional slices from the top and bottom, leaving the ingot surface intact (unscarfed). A second set of samples was taken adjacent to the first following machining 6.4 mm (0.250 in) from the diameter of the ingot. The purpose of these (scarfed) ingot samples was to determine whether the VAR or ESR practice was effective in concentrating the oxides on the remelted ingot surface skin.

The samples taken from the VAR and ESR ingots were subjected to the same procedure for oxide extraction and filtration as was used for the VIM heats described previously. The results are summarized in Table 4.

Table 4. Gravimetric Results of the VAR and ESR Remelt Study

Melt No.	Description	Residue Weight (mg) "A" Layer	Residue Weight (mg) "B" Layer	Approx. PPM Oxide
11	VIM Heat	2.4	0.3	1.5
12	VAR - 0.64 Kg/min Scarfed	8.5	0.3	5
13	VAR - 0.64 Kg/min Unscarfed	2.2	0.4	1
14	VAR - 1.1 Kg/min Scarfed	2.7	0.2	2
15	VAR - 1.1 Kg/min Unscarfed	2.4	0.2	2
16	VAR - 1.9 Kg/min Scarfed	2.4	0.2	2
17	VAR - 1.9 Kg/min Unscarfed	4.1	0.3	2
18	ESR - Scarfed	0.6	0.1	<1
19	ESR - Unscarfed	0.9	0.3	<1

RESULTS AND DISCUSSION

Cleanliness Evaluation Technique

The gravimetric results for the VIM crucible study (Table 3) reveal a substantially higher residue weight for the "A" layer extractions compared to the "B" layer extractions. This indicates the effectiveness of the floatation process resulting from EB remelting. Analyses of selected residues from the

"A" layers revealed that 85-93% of the residues are oxides. Similar analyses of "B" layer residues revealed less than 30% of the residues to be oxides and those which were present were of a substantially smaller size than those for the "A" layer. Thus, it can be concluded that the "A" layer residue weights are representative of the oxide content of the original sample. An estimate of approximate PPM oxide content was calculated by dividing the "A" layer weight by the initial sample weight. The resultant values (Table 3) show good correlation with the area measurements of the oxide rafts on the EB remelted ingots.

Ingot Evaluation

From the results presented in Table 3, it can be seen that the three heats of material produced in zirconia crucibles (Heat # 8, 9, 10) consistently resulted in the highest concentration of oxides with values ranging from 8 ppm to 9 ppm. Alumina rammed crucibles resulted in the second highest inclusion content of 7 ppm (Heat #2) and 6 ppm (Heat #7) although the high purity alumina preform crucible (Heat #1) resulted in a relatively low oxide content of 1 ppm. The high cost and poor thermal shock resistance of this crucible would preclude it from serious consideration for production useage. The magnesia preform crucible resulted in a relatively high oxide concentration of 6 ppm and also exhibited poor thermal shock resistance. However, the magnesia rammed crucibles resulted in the lowest inclusion contents with values of 4 ppm and 1 ppm for the new and used crucible, respectively. The MERL 76 heat which was melted without the reactive element additions resulted in by far the lowest level of inclusions (0.2 ppm).

Examination of the oxide residues for the VIM crucible study indicated the predominant oxide to be hafnia (HfO_2) for all MERL 76 heats independent of crucible composition, with HfO_2 contents ranging in excess of 90% of the extracted residue. Minor quantities of alumina were also found with lesser amounts of silica. The oxide species for the MERL 76 less reactive element heat could not be identified. For the MERL 76 alloy composition, it appears that melt reactions between hafnium and dissolved oxygen in the melt and/or oxides in the crucible represent the predominant source for oxide contamination of the ingot as opposed to crucible erosion/spallation.

The results of the VAR and ESR remelt studies are shown in Table 4. The oxide content of the starting VIM heat produced in magnesia crucible was calculated at 1.5 ppm. Values obtained following VAR under the various melt rates investigated

ranged from 1 to 2 ppm for both scarfed and unscarfed samples with the exception of the scarfed ample melted at 0.64 Kg/min (#12) which showed an unexplainable higher oxide content of 5 ppm. From the overall results of the VAR evaluation, it does not appear that oxide content is altered by any appreciable amount by this remelt technique nor does it appear to concentrate oxides on the ingot surface. The ESR ingot appeared to yield a significantly lower inclusion content of 0.3-0.6 ppm. Analysis of the residues revealed that for both the VAR and ESR ingots, hafnia represented the major oxide species with minor amounts of alumina also detected.

CONCLUSIONS

1) Magnesia based crucibles appear to result in lower oxide concentrations in MERL 76 VIM ingot followed by alumina based and zirconia based crucibles.

2) Hafnia represents the dominant oxide species in MERL 76 VIM ingot independent of the composition of the crucible.

3) Vacuum arc remelting appears to have little or no effect on ingot oxide content while electroslag remelting resulted in significnatly reduced oxide content.

4) The evaluation technique developed for determination of oxide contents in ingot appears to offer a useful tool for rating ingot cleanliness.

REFERENCES

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