COMPOSITIONAL CONTROL AND OXIDE INCLUSION LEVEL COMPARISON OF PYROMET®718

AND A-286 INGOTS ELECTROSLAG REMELTED UNDER AIR VS. ARGON ATMOSPHERE

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Summary

The effect of electroslag remelting (ESR) Pyromet 718 and A-286 alloys under an argon atmosphere and its resulting effect on ingot compositional control and oxide inclusion level were investigated using a laboratory consumable electrode furnace. Chemical composition data indicate that an argon cover during ESR rendered the slag a closed system where oxidation/reduction reactions were interdependent. This result provides potential for improved compositional control and lower ingot oxygen levels compared to air-ESR. Reasonable correlation between ingot oxygen content and volume percent inclusion level was attained and data show that low oxygen argon-remelted ingots exhibited a lower inclusion content than air-remelted ingots of higher oxygen content. These preliminary results demonstrate the improved process control of slag reactions provided by argon-ESR has the potential to improve the compositional control and "cleanliness" of superalloy ingots and consequently their mechanical properties.

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Introduction

Melting processes that can provide precise compositional control, superior "cleanliness," and control of solidification structure are of great interest as pressure mounts to improve the mechanical property performance of superalloys in critical applications. Electroslag remelting (ESR) is a common refining method, but can encounter difficulty in controlling ingot composition of superalloys containing high levels of reactive elements (Ti,Al) due to molten metal/slag and slag/air chemical reactions. In addition, if proper slag deoxidation practices are not followed, ingot oxygen levels can increase. The use of an argon cover over the slag bath during ESR to improve process control over slag reactions was investigated and the results are presented.

Experimental Procedure

Six inch (150 cm) rd electrodes of Pyromet 718 and A-286 were electroslag remelted into eight inch (200 cm) rd ingots in Carpenter Technology's laboratory consumable electrode furnace. For comparison, melting trials were conducted under both air and argon atmospheres. During argon remelting, the exit gas v/o oxygen content from the melting chamber was maintained at less than 2%. Pyromet 718 ingots were remelted at $_{\rm V}215$ lb/h using an "A" slag of 36% CaF2, 29% CaO, 3% MgO, and 32% Al2O3. Pyromet A-286 ingots were remelted at $_{\rm V}150$ lb/h using a "B" slag of 32% CaF2, 30% CaO, 3% MgO, 34% Al2O3, and 1% SiO2. Melting parameters for 718 ingots (1, 2, 3) and A-286 ingots (4, 5, and 6) were:

Electrode composition was determined at top and bottom locations prior to remelting while remelted ingot compositions were determined from A, B, C and X locations as diagrammed in Figure 1. Oxygen analyses were determined via a combustion infrared detection method with a precision of \pm 5 ppm at levels below 40 ppm and \pm 10 ppm at levels above 40 ppm. Sulfur levels were measured with a \pm 1-2 ppm level of precision.

Metallographic specimens adjacent to compositional analysis specimens from the center of the A, B, C, and X disc locations of each remelted Pyromet 718 and A-286 ingots were removed to measure oxide inclusion content. Specimens were hot isostatically pressed (HIP'd) at 2125°F/3h/15ksi to eliminate shrinkage porosity from solidification. Each sample was automatically polished and rated for oxide inclusion content on three parallel planes using a Leitz TAS quantitative image analysis microscope. An average of 200 fields were rated to generate each data point. Image analysis used a screen magnification of 2620x and determined volume percent oxide content, average particle diameter, and average particle area. Confidence limits of $\pm 95\%$ also were determined by the microscope for each data point. The mean (\bar{x}) and standard deviation (s)for the inclusion content of each ingot were calculated and levels of significant difference between mean volume percent inclusion levels of the respective ingots determined. Linear correlation coefficients (r) between volume percent inclusion content and oxygen content were also calculated. SEM analysis was performed on randomly selected primary carbides containing

TABLE I

SELECTED PYROMET 718 AND A-286 ELECTRODE
AND INCOT ELEMENTAL COMPOSITIONS

Pyromet 718 Ingot 1 (Air + 6% TiO ₂ + Al Deoxidant Addition)							Pyromet A-286 Ingot 4 (Air + 3% TiO ₂)						
	X	A	X	С	В	A		X	A	X	C	В	A
S (PPM)	10	20	7	7	7	7	Si	0.22	0.22	0.27	0.23	0.22	0.23
Ϊi	0.94	1.00	0.94	0.96	1.00	1.07	S (PPM)	<10	<10	<5	<5	<5	<5
Al	0.58	0.62	0.45	0.50	0.50	0.48	Τi	2.02	2.15	1.81	1.62	1.78	2.02
O (PPM)	<5	<5	33	29	38	46	A1	0.23	0.23	0.29	0.30	0.22	0.25
							O (PPM)	11	<5	43	35	34	22
		Ingot 2	(Argon)			Ingot 5 (Argon)						
S (PPM)	10	40	<5	<5	5	6	Si	0.28	0.22	0.29	0.26	0.23	0.23
Ti	1.02	1.00	0.81	0.87	0.95	1.07	S (PPM)	<10	<10	<5	<5	<5	<5
Al	0.56	0.58	0.65	0.63	0.60	0.51	Τi	2.01	2.06	1.67	1.81	1.95	2.19
D (PPM)	<5	<5	13	20	24	8	Al	0.28	0.31	0.50	0.46	0.42	0.27
							O (PPM)	<5	<5	7	9	10	5
	Inq	ot 3 (A:	rgon + i	s≰ TiO_)		Ingot 6 (Argon + 6% TiO_)						
Ingot 3 (Argon + 6% T10 ₂)										•	2		
S (PPM)	10	10	7	8	9	8	Si	0.18	0.19	0.31	0.24	0.23	0.23
Ti	1.01	1.02	0.94	0.98	0.97	1.08	S (PPM)	NA	NA	<5	<5	8	<5
A1	0.53	0.54	0.54	0.56	0.57	0.49	Ti	2.02	2.13	1.87	2.03	2.08	2.24
0 (PPM)	22	<5	12	15	20	<5	Al	0.35	0.34	0.41	0.35	0.36	0.28
							O (PPM)	NA	NA	<5	5	10	<5
All Pyromet 718 ingots remelted utilizing a 36% CaF ₂ , 29% CaO, 3% MgO, 32% Al ₂ O ₃ slag.							All Pyromet A-286 ingots remelted utilizing a 32% CaF_2 , 30% CaO_2 , 3% MgO, 34% Al_2O_3 , 1% SiO_2 slag.						

All values are weight percent

TABLE II

PYROMET 718 AND A-286
OXIDE INCLUSION DIAMETER AND AREA DATA Ingot Top Diameter (um) (Area (um²) Mean (X) Std. Dev. (s) Ingot Mean (\overline{X}) Std. Dev. (s) Disc 1 0.978 0.138 0.949 0.398 2 0.945 0.154 0.866 0.267 Disc 25" 1.001 0.161 1.107 0.384 "C" Disc 0.736 0.128 0.508 0.189 Disc 0.569 0.056 0.364 0.205 Ingot Bottom 0.690 0.113 0.482 0.171

<u>Figure 1</u> - Ingot Disc Locations Sampled for Argon and Air ESR Study

NA - Not analyzed

inclusions from the 3-C and 6-A ingot locations. Visual metallographic inspection of the specimens was performed and electron microprobe analysis performed on three isolated inclusions observed at the 3-X ingot location.

Experimental Results

The nominal analysis (w/o) of the VIM Pyromet 718 and arc-AOD A-286 electrode material used in this study was as follows:

Pyromet A-286 0.030 0.35 0.22 0.015 0.001 14.50 24.60 1.10 0.24 ---- 2.05 0.30 0.005 Bal.

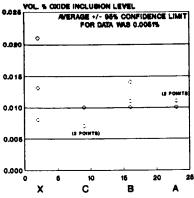
Table I lists the elemental levels which were altered by remelting for Pyromet 718 ingots 1, 2, and 3. Data of ingot 1 (air cover) show Al decreased 0.10-0.15% from electrode levels and oxygen increased from <5 ppm to as high as 46 ppm. A slight increase in Ti was also observed at the ingot top. Sulfur was lowered a slight amount by remelting. Ingot 2 (argon cover) revealed an overall loss of Ti, but increase in Al. The largest loss of Ti and highest increase in Al was at the X location, but this difference diminished as remelting progressed until there was a slight increase of Ti and loss of Al observed at the A location. A smaller increase in oxygen content was observed compared to ingot 1. Sulfur level was also lowered. Ingot 3 (argon cover + TiO_2 slag addition) showed much improved compositional control compared to ingot 2. Only a slight loss of Ti and increase in Al were observed. Oxygen levels were comparable to ingot 2. Sulfur level also remained low.

Table I also lists the elemental levels that were altered by remelting of Pyromet A-286 ingots 4, 5, and 6. Ingot 4 (air) shows a significant loss of Ti at all locations during remelting and a slight increase of Al. Oxygen level increased significantly at all locations during melting while sulfur was maintained at low levels and Si was essentially unchanged. Ingot 5 (argon) also showed a significant overall loss of Ti during melting, but with a corresponding increase in Al content. The condition was most pronounced at the X location and diminished as remelting progressed until a slight increase in Ti was observed at the A location. Oxygen content increased only a slight amount compared to ingot 4. Sulfur remained at very low levels and Si level was basically unchanged. Ingot 6 (argon + 6% TiO₂) showed a significantly smaller Ti loss and Al increase compared to ingot 5 although a slight increase of Si was observed. Oxygen and sulfur levels were maintained at very low levels.

Figure 2 lists the volume percent inclusion data for ingots 1-6. Given the very low measured volume fractions, scatter in the data is minimal for a specified location. Table II lists the average particle diameter and area of the inclusions and shows that averages for both Pyromet 718 and A-286 were in the vicinity of 1 micron or less. Correlation coefficients (r) between volume percent inclusion content and oxygen content for the respective Pyromet 718 and A-286 data were calculated and determined to be 0.769 and 0.673 respectively.

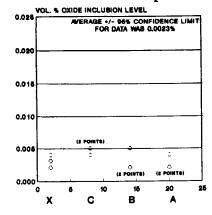
Image analysis showed the great majority of inclusions to be located within Ti-rich carbides and/or carbonitrides. Figures 3 and 4 show respective SEM photomicrographs of a carbide cluster and isolated carbide containing inclusion nuclei from the 3-C ingot location. For the inclusion shown in Figure 4, energy dispersive spectrographic (EDS) analysis with the SEM indicated the carbide/inclusion center to be very high in Ti with significant amounts of Al and Mg while an analysis of the outer shell

718 INGOT #1 (AIR + 6%TIO, + AI DEOX)



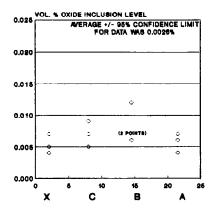
INGOT HEIGHT (IN) AND LOCATION

A-286 INGOT #4 (AIR + 3%TIO₂)



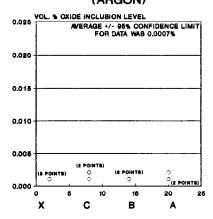
INGOT HEIGHT (IN) AND LOCATION

718 INGOT #2 (ARGON)



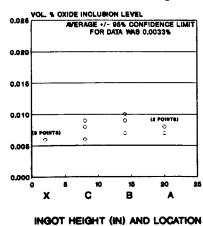
INGOT HEIGHT (IN) AND LOCATION

A-286 INGOT #5 (ARGON)

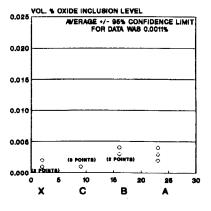


INGOT HEIGHT (IN) AND LOCATION

718 INGOT #3 (ARGON + 3% TiO₂)



A-286 INGOT #6 (ARGON + 6% TiO₂)



INGOT HEIGHT (IN) AND LOCATION

Figure 2 - Volume Percent Inclusion Data for Laboratory Pyromet 718 and A-286 $8^{\prime\prime}$ Rd. Ingots Electroslag Remelted under Argon and Air Atmosphere

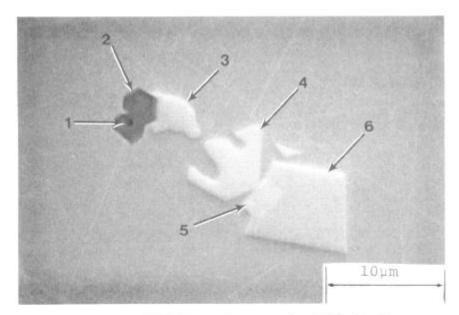


Figure 3 - SEM Photomicrograph of Typical Primary Carbide Cluster Observed In Pyromet 718 (Ingot 3-C Location). Note Al-Mg-Rich Inclusion (1) Located Within Ti-Rich Carbide (2). Columbium-Rich Carbides Constitute Remainder of Cluster (3,4,5,6).

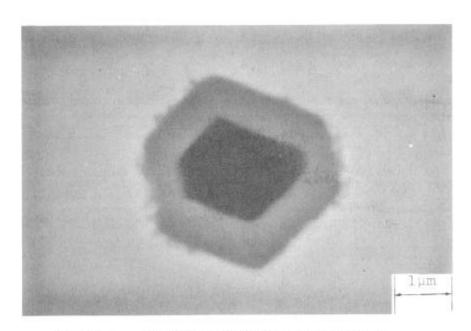


Figure 4 - SEM Photomicrograph of Ti-Rich Primary Carbide Containing Al-Mg-Rich Inclusion Nucleus (Ingot 3-C Location).

revealed only very high Ti levels. Analysis of a randomly selected primary carbide in A-286 with an inclusion nucleus showed it to be very high in Ti with small levels of Al and Mg. In both 718 and A-286, inclusions were found only at the center of Ti-rich primary carbides. However, in both 718 and A-286, Ti-rich carbides were frequently observed within larger Cb-rich carbides. Microstructural analysis also showed inclusions were also

more frequent in 718 than in A-286 and more frequent in air remelted ingots than argon remelted ingots. Metallographic observation also revealed three small inclusions enriched in Al, Ca, and Mg at the X location of ingot 3.

Discussion

Compositional Control

Examination of Pyromet 718 ingots 1, 2, and 3 revealed important insights into slag reactions and their effect on ingot composition during ESR. Ingot 1 demonstrated that even with ${\rm TiO_2}$ and A1 deoxidation slag additions, a significant loss of A1 occurred. Further refinement of slag compositions through ${\rm TiO_2}$ and deoxidant additions can provide improved ingot compositional control as is evidenced by routine air remelting of 718 production ingots. However, the increased oxygen content of ingot 1 compared to the electrode content indicated that even with a slag deoxidation addition of A1, the ESR operation increased the ingot oxygen content. The loss of Ti and increase in A1 in ingot 2 demonstrates that the argon cover over the slag during ESR isolated it from the air and rendered the slag a closed system where the slag oxidation/reduction reactions became interdependent. Similar behavior was observed in Ti stabilized stainless steels by Schwerdtfeger et al.(1). The Ti and A1 compositional variations can be explained by the reaction:

$$3\text{Ti} + 2(\text{A1}_2\text{O}_3) \rightleftharpoons 4\text{A1} + 3(\text{TiO}_2)$$
 (1)

as discussed by Pateisky et al(2). This reaction occurred as the slag attempted to reach an equilibrium state during remelting. The interrelationship between Ti and Al and low ingot oxygen level also suggest that the oxidation/reduction reactions of Ti and Al with FeO were minimized and that low slag oxygen potentials were obtained during ESR under argon(1,3). In argon-ESR, the isolation of the slag from an air atmosphere and the interdependence of slag reactions favors a low slag oxygen potential which consequently produces a remelted ingot of lower oxygen content compared to air-ESR (compare ingots 1 and 2)(4). Data of Ingot 3 demonstrated that when a slag of near equilibrium composition was used in conjunction with an argon cover during ESR, improved control of ingot Ti and Al levels and lower ingot oxygen levels can be obtained compared to ingots remelted via air-ESR. Remelting under an argon cover also maintained very low sulfur levels and did not adversely affect the desulfurizing capability of the slag.

The A-286 data of ingots 4, 5, and 6 essentially confirmed the trends observed in Pyromet 718 heats, but also revealed that control of ingot composition can be extremely difficult during air-ESR of alloys with a high Ti/Al ratio as shown by ingot 4. Ingot 4 also shows the substantial increase of ingot oxygen content after air-ESR just as did Pyromet 718 ingot 1. Ingot 5 (argon) data reflect the reduction of Al_2O_3 in the slag to form TiO₂ as the slag attempted to reach an equilibrium state. Ingot oxygen content only increased a slight amount after remelting under an argon atmosphere. Ingot 6 (argon + 6% TiO $_2$ slag addition) data showed improved compositional control of Ti and Af compared to ingot 5 while maintaining a low ingot oxygen content. Utilization of slightly higher TiO₂ content in the slag during ESR under argon should improve compositional control of Ti and Al while maintaining low ingot oxygen levels. Comparison of ingots 3 and 6 demonstrate that the proper slag ${
m Ti0}_2$ content to maintain compositional control will vary according to alloy composition, particularly Ti and Al content(2). This demonstrated that the argon-ESR process is capable of remelting Ti-Al containing superalloys with improved compositional control while maintaining low ingot oxygen and sulfur levels.

Ingot Inclusion Level

An important implication of the lower ingot oxygen content obtained through argon-ESR is its effect on ingot inclusion content. A comparison of the calculated inclusion volume percent averages showed that the difference between the two 718 ingots remelted in argon and air was significant above a 95% confidence level. This trend was also observed in the A-286 ingots. The statistically significant lower inclusion levels of the argon remelted heats indicated that the lower oxygen content produced a direct reduction in the inclusion levels. The three small Al-Ca-Mg-rich inclusions at the X location of ingot 3 can be attributed to entrapped slag from the start up of the melt at that location and do not indicate a refining problem with the slag.

Figure 5 shows bar charts where the average of the three measured inclusion levels are compared with the measured oxygen content for each location. Previous investigations(5,6) have found reasonably good correlations between oxygen level and oxide inclusion content for oxygen levels above 40 ppm. Given both the low measured oxygen levels (precision of \pm 5-10 ppm) and the very low measured inclusion levels, the degree of correlation of this study is considered quite good.

It is interesting to note that for a given oxygen level, the inclusion level of Pyromet 718 ingots is higher than that of A-286. Factors contributing to this difference could include both varying oxygen solubilities of the 718 and A-286 matrices and varying densities of the oxide inclusions depending upon the oxide formed. Microstructural analysis also showed that lower oxygen levels in argon remelted ingots did reduce the number of inclusions compared to air remelted ingots. Therefore, the data indicate that a given oxygen level can produce varying inclusion levels depending on the alloy system and oxide species formed during melting and solidification.

Work by Fox et al. (7) on alloy 718 also showed that Ti-rich carbides in 718 nucleated on small aluminum oxide inclusions with smaller amounts of Ca and Mg also detected. Detection of Al and Mg at the center of Ti- rich carbides in both 718 and A-286 supports this work and indicates that A1 and Mg oxide inclusions were nucleation sites for Ti-rich carbide precipitation during solidification. Thus, if the inclusion level is lowered, it is quite possible that the average size of Ti-rich carbides could be reduced since their nucleation could be delayed during solidification. Mitchell and Tripp (8) have addressed the potential deleterious effect which TiN/oxide clusters can exert on superalloy mechanical properties. This topic is of considerable importance since a uniform primary carbide distribution imparts optimum mechanical properties to an alloy component. The preliminary data of this study suggest argon-ESR has the potential to achieve oxygen levels of < 10 ppm in superalloy ingots and consequently very low oxide levels. This could contribute to improved carbide distributions by minimizing the interaction between TiN's and oxides and improve alloy performance. The potential for improved alloy "cleanliness," and more uniform carbide distributions makes the argon-ESR process worthy of continued investigation. Also, further refinements in slag composition and deoxidation/desulfurization additions could produce very "clean" ingots with mechanical properties superior to either conventional VAR or air-ESR ingots.

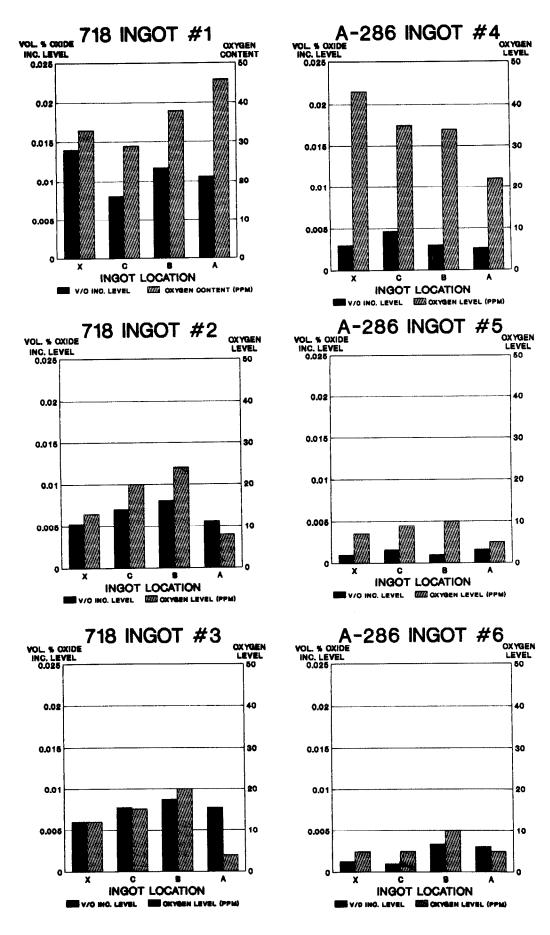


Figure 5 - Comparison of Oxygen and Average Volume Percent Inclusion Levels in Laboratory Pyromet 718 and A-286 $8^{\prime\prime}$ Rd. Ingots Electroslag Remelted under Argon and Air Atmosphere

Conclusions

- 1. Argon-ESR effectively isolated the slag from the air atmosphere. This condition caused slag oxidation/reduction reactions to become interdependant while also lowering the oxygen potential of the slag. This resulted in improved control of the ingot Ti, Al levels and lowered the oxygen content of the ingot.
- 2. Oxygen content and measured volume percent inclusion level demonstrated reasonable correlation.
- 3. Low oxygen argon-remelted ingots demonstrated lower inclusion levels than air-remelted ingots of higher oxygen content. Low inclusion levels may also help reduce the primary carbide size and assist in minimizing carbide clustering.

Acknowledgements

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