

PRODUCTION OF HIGH-STRENGTH P/M DISC ALLOYS

BY "SUPERCLEAN" CAST/WROUGHT TECHNOLOGY

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Abstract

Alloy APK12, which corresponds to the composition of UDIMET* alloy 720 has been produced by conventional powder metallurgy techniques as well as Electron Beam Cold Hearth Refining (EBCHR), which is a cast/wrought method of producing "superclean" alloys. A comparison of chemistries, cleanliness levels, macro and microstructures, hot workability and mechanical properties, especially low cycle fatigue lives, has been carried out to ascertain the benefits of EBCHR.

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Introduction

Conventional atomization techniques of producing segregation prone, high strength nickel-base superalloys for critical rotating turbine disc applications suffer from the inherent danger of contamination during powder handling. This, added to the high cost of manufacturing material by this route, is persuading both alloy producers and engine builders to investigate alternative melting methods. Traditional methods such as Electroslag and Vacuum Arc Remelting (ESR and VAR) have been incorporated into "triple melt" routes such as VIM + ESR + VAR which at present are the accepted cast/wrought techniques for producing these alloys. Cleanliness levels obtained by these routes are still no better than those achieved using powder metallurgy (P/M). Refractory-free melting systems such as Electron Beam Cold Hearth Refining (EBCHR), which are currently the most advanced techniques for producing "superclean" material, have therefore been seriously assessed. The highly flexible and manoeuvrable EB guns allow excellent control of inclusion removal, thus improving cleanliness, as well as minimising segregation by regulating ingot solidification.

A significant amount of work has been carried out on wrought alloys such as INCONEL⁺ alloy 718 with regard to the macro and microstructure as well as the mechanical properties of the cold hearth refined (CHR) material (1, 2, 3). Comparisons between CHR and conventionally melted VIM + ESR and VIM + VAR material has also been reported (4). Much higher strength materials, which are by their very nature more sensitive to defects, stand to benefit more from the cold hearth refining. The work reported in this paper has therefore concentrated on Alloy APK12, which is similar in composition to UDIMET alloy 720.

Melting of Alloy APK12 by EBCHR

EBCHR is essentially a process whereby feedstock material is melted in a high vacuum chamber by electron beam guns into a water cooled copper hearth. Low density inclusions such as oxides and nitrides are allowed to rise to the surface in the hearth and are then either swept back by the beam and/or physically restrained by a mechanical barrier from reaching the solidifying ingot at the other end. The EBCHR process and its refining effects are discussed in detail elsewhere (5).

The feedstock used was extruded powder Alloy APK12 which has a nominal composition (in weight %) of 0.03 C, 18 Cr, 15 Co, 3 Mo, 1.25 W, 5 Ti, 2.5 Al, 0.035 B and 0.035 Zr. The powder was atomised using argon and sieved, again under argon, using -150 μ m mesh to obtain a maximum particle size of 150 μ m. It was then filled into mild steel cans under vacuum and extruded to 130 mm diameter bars. After decanning, these bars were used for both remelting by EBCHR and as the standard comparison material against the "superclean" Alloy APK12.

EBCHR ingots of 250 mm diameter and weighing approximately 450 kg were melted (figure 1). A thorough structural and chemistry examination was conducted which essentially showed that no unacceptable macrosegregation occurred and that oxygen and nitrogen contents were significantly reduced, indicating reduced numbers of oxides and nitrides. The oxygen level (down from 65 ppm in feedstock to 12 ppm after EBCHR), which is the more important element due to the particularly detrimental effects on properties of large oxides, is especially encouraging as it is close to its solubility level at the superheats used. Therefore the amount of free oxygen available to form oxides is negligible. The chromium content is reduced from 17.67% in the feedstock to 17.05% in the EB refined alloy. Chromium evaporation is critically dependent upon heat input levels and can therefore be controlled

to some extent. Lower heat inputs also mean lower superheats and therefore lower solubility levels for both oxygen and nitrogen. Low power inputs onto the solidifying ingot surface are also beneficial in terms of structure as finer grain material is obtained (6).

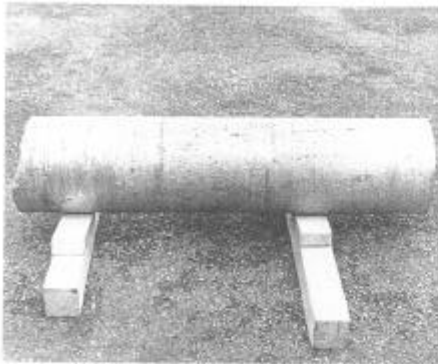


Figure 1 - EBCHR Alloy APK 12 ingot



Figure 2 - P/M Alloy APK 12 button on left showing large inclusion cap compared to EBCHR button on right

Cleanliness of EBCHR and P/M Alloy APK12

Electron Beam Button Melting (EBBM) (7) techniques were used to determine the cleanliness of Alloy APK 12 produced via the two different routes. As figure 2 shows, on a macro scale, the cap of inclusions obtained on a button of the extruded powder feedstock is considerably larger than that on the EBCHR Alloy APK12. When examined on the Scanning Electron Microscope, it is evident that the caps on both buttons almost exclusively consist of a monolayer of titanium nitrides (figure 3). The reduction in the cap size on the EBCHR Alloy APK 12 button is therefore a direct consequence of the fall in nitrogen content from 47 ppm in the feedstock to 35 ppm after cold hearth refining. On the whole the nitrides are extremely small ($<5 \mu\text{m}$) and therefore of no great concern as long as they do not agglomerate and form clusters. The inclusions of real interest are the oxides, which can have particularly adverse effects on properties such as low cycle fatigue (LCF) life. It is generally accepted that oxide particles greater than $50 \mu\text{m}$ in size are undesirable in high strength superalloys. There is every possibility, however, that defects of this size, and indeed up to $150 \mu\text{m}$, will be found in powder produced to -150 mesh. It is not inconceivable that inclusions larger than this size will survive the sieving process if they are of the right shape, eg long and thin needles. Defects of this size are not detectable by traditional ultrasonic techniques and therefore EBBM is a very useful tool for assessing the relative cleanliness of a fairly large sample (approx. 1 kg) with regard to these types of inclusions.

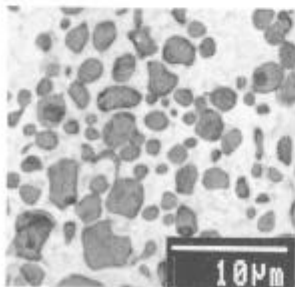


Figure 3 - Monolayer of Ti nitrides

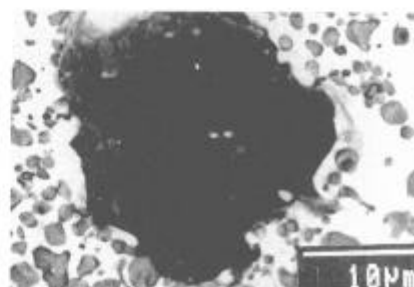


Figure 4 - $100 \mu\text{m}$ oxide on P/M button (left) and $25 \mu\text{m}$ oxide on EBCHR button (Right)

It can be seen from figure 4 that the largest oxides found on the EBCHR Alloy APK12 button were 25 μm in size. In the case of the extruded powder feedstock, however, particles up to 100 μm were evident. In both cases they were primarily rich in aluminium with traces of magnesium, silicon and calcium. On average there were only around eight oxide particles in the 20 - 25 μm size range on a button of the EBCHR material whereas there were ten particles in the 80 - 100 μm size range on a button of the P/M Alloy APK 12. This clearly demonstrates that the EBCHR process had succeeded in eliminating the large oxide defects which are particularly detrimental to critical mechanical properties in high strength disc superalloys such as UDIMET alloy 720.

Processing EBCHR Alloy APK12 by Cast/Wrought Techniques

The primary reason for processing Alloy APK12 via powder metallurgy is its propensity to segregate if melted in ingot form using traditional ESR and VAR techniques. This is because it is highly alloyed and therefore extremely sensitive to solidification rates. In both ESR and VAR these cannot be controlled to any great extent except by reducing the ingot diameter, which ensures quicker local solidification rates. In EBCHR however, because the primary direction of heat extraction is through the top surface of the ingot, and as the heat source is so controllable, the solidification rate can be regulated to a high degree. The macrostructure can therefore be optimised to give a desired grain structure by altering the power input onto the solidifying ingot (6). This makes the material more amenable to hot working.

There is currently a trend in the P/M industry towards compacting the powder using extrusion rather than hot isostatic pressing. There is evidence that thermomechanical processing (TMP) significantly reduces and disperses hard defects thereby improving the LCF properties of nickel base P/M superalloys (8). TMP also refines the grain size allowing enhanced ultrasonic inspectability. Extrusion was therefore used for compacting the -150 μm Alloy APK12 powder as well as hot working the EBCHR material. In both cases 4" diameter machined billet was produced for isothermally forging to discs. An extrusion ratio of 5:1 was obtained. The billets were isothermally forged at 1107°C to a 70% reduction into small helicopter disc shapes (figure 5). Both materials forged very well without any cracking and as figure 6 shows an extremely uniform fine grain macro structure was obtained throughout the discs. On a micro level, however, there were occasional grains as large as ASTM 5 amongst the ASTM 11 - 12 average grain size measured in the EBCHR Alloy APK12 disc (figure 7). These larger grains had survived the isothermal forging in an unrecrystallised state. They were originally apparent in the as-extruded microstructures, most probably due to the un-optimised TMP conditions. Similarly some porosity was evident in the P/M Alloy APK12 (figure 8). This porosity was not observed in the as-extruded material and



Figure 5 - EBCHR forged disc

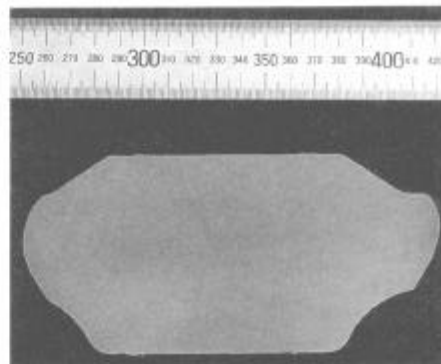


Figure 6 - Fine grain macrostructure

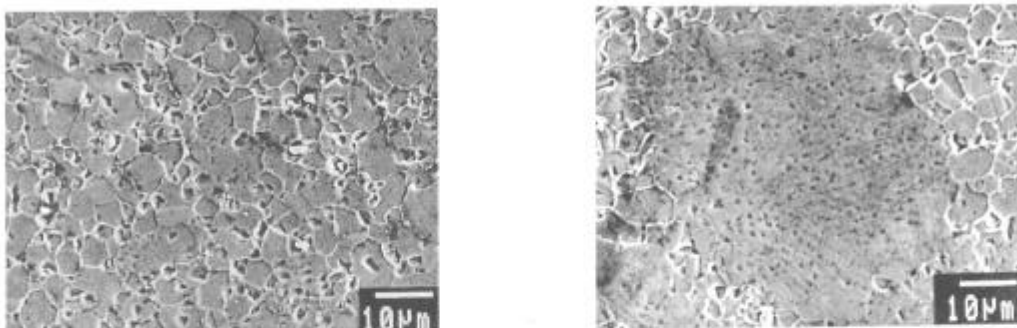


Figure 7 - Typical ASTM 11-12 grain size (left) with isolated large grain (right) in EBCHR Alloy APK 12

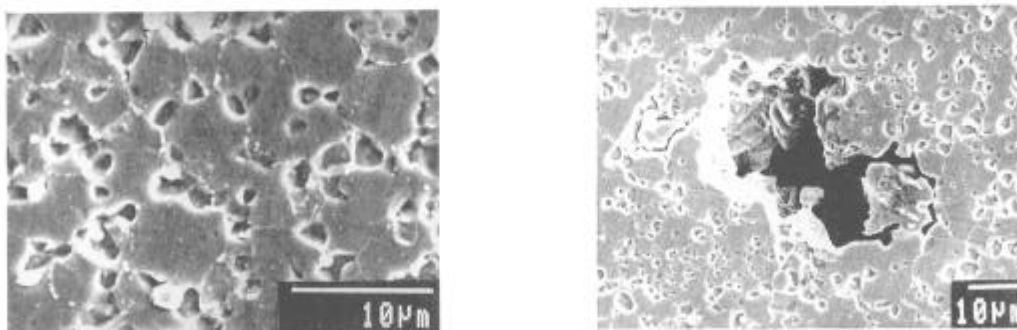


Figure 8 - Typical ASTM 11-12 grain size (left) with example of porosity (right) in forged P/M Alloy APK 12

the argon analysis gave a content of only 1.2 ppm, indicating that high gas content was not the cause. A combination of strain/thermally induced response was most likely the cause of the porosity. The discs were given the standard heat treatment of 4 hours 1100°C oil quench + 24 hours 650°C air cool + 16 hours 760°C air cool. In both cases extremely fine intragranular γ' with some coarse intergranular γ' was obtained. There was also grain boundary precipitation of Cr_{23}C_6 , TiC and $(\text{Cr, Mo, W})_3\text{B}_2$ in both the variants.

Mechanical Properties of EBCHR and P/M Alloy APK12

The isothermally forged discs were heat treated as detailed above and a mechanical property evaluation was performed on this material.

Tensile Strengths

Tensile test specimens were taken in the tangential direction from the forgings and were tested at both room temperature and 650°C. Duplicate tests were performed and the average ultimate tensile strengths, 0.2% yield strengths and the elongations are presented in table 1. The results show

Table I Tensile Properties of P/M and EBCHR Alloy APK 12

MATERIAL	TEST TEMP C	STRESS N/mm2		ELONG %
		0.2% P.S.	UTS	
P/M ALLOY APK12	20C	1,212	1,595	14
	650C	1,100	1,474	16
EBCHR ALLOY APK12	20C	1,161	1,512	9
	650C	1,111	1,496	12

that both the tensile and yield strengths at room temperature of the P/M Alloy APK12 are slightly higher than the EBCHR version. At 650°C however, the latter performs slightly better, although its elongation is once again poorer.

Fatigue Crack Propagation Results

Duplicate fatigue crack growth rate tests were conducted on compact specimens in air at 600°C, R-ratio = 0.1 and a frequency of 15 cpm trapezoidal waveform for both versions of Alloy APK12. The crack growth was monitored using a Direct Current Potential Drop method. The results are shown in figure 9. The crack growth characteristics of both versions of the alloy are essentially similar. This is to be expected as crack propagation rates primarily depend upon the basic microstructural features such as grain size rather than the cleanliness, and as both the P/M and EBCHR materials had an average grain size of ASTM 11 - 12, the crack growth rates were comparable. The fracture path was transgranular in both cases.

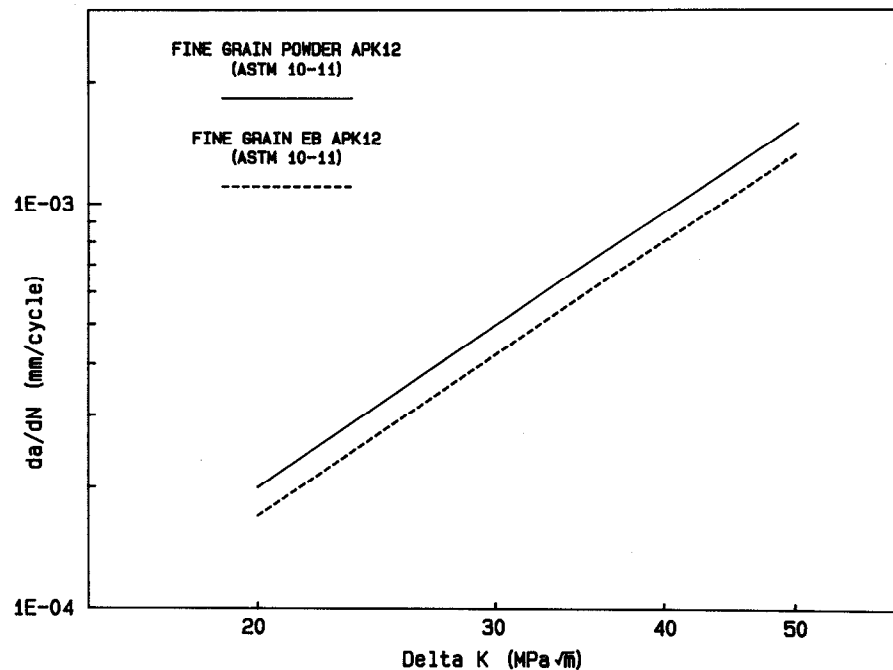


Figure 9 - Fatigue crack growth curves showing essentially comparable properties for both P/M and EBCHR Alloy APK12

Low Cycle Fatigue Lives

An extensive programme of LCF property evaluation was conducted on the isothermally forged discs to establish whether the improved cleanliness of the EBCHR Alloy APK12 was "cashable" in terms of better lives. The tests were carried out in air at 600°C using a frequency of 1 Hz (sinusoidal waveform) under a load control regime.

Stress vs Cycles to Failures (S-N) curves were initially constructed (figure 10). It can be seen that the curve for the EB refined Alloy APK 12 has been significantly shifted to the right indicating an improvement in LCF life at all stress levels. The reduction in the number and size of low density inclusions has obviously contributed to this increased life. To demonstrate the consistency and homogeneity of this improvement in cleanliness, one

testing condition (1060 MPa maximum stress) was picked to assess the scatter obtained over 12 samples. As figure 10 shows, there is reduced scatter obtained in the EBCHR material compared to the P/M Alloy APK12. Whereas for the powder material the difference between the shortest and longest lives is well over an order of magnitude, in the case of the EB refined material it is considerably less than this (table 2). As well as the reduced scatter, indicating a greater consistency of cleanliness, the lives of the EBCHR alloy samples were considerably longer (an average life of 143650 cycles compared to 38224 cycles).

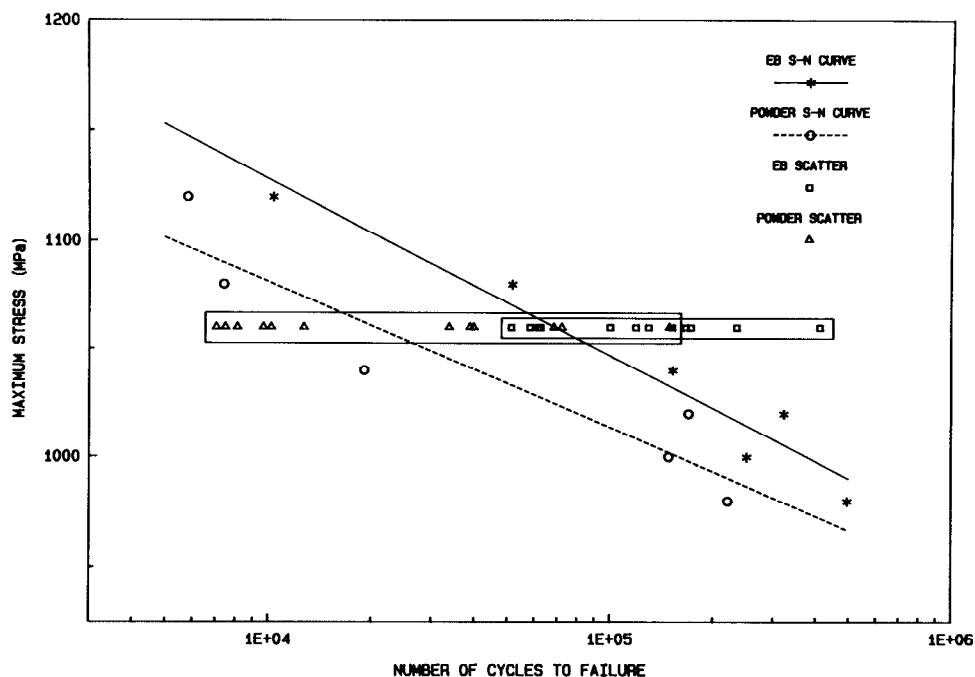


Figure 10 - S-N Curves and scatter at one condition showing significant improvement in LCF lives for the EBCHR Alloy APK 12.

Table II Scatter results at 1060 MPa

CYCLES TO FAILURE AT 0-1060 MPa	
EBCHR ALLOY APK12	P/M ALLOY APK12
172, 674	7, 494
61, 718	9, 679
412, 236	39, 987
119, 425	68, 627
62, 659	7, 050
129, 995	10, 209
152, 242	38, 975
51, 525	33, 905
100, 380	8, 132
58, 472	72, 398
235, 283	12, 726
167, 188	149, 507

An examination of the fracture surfaces of 18 samples (6 from S-N curve plus 12 scatters) from each version of the alloy was made to ascertain the initiation modes. Essentially four types of initiation sites were identified:

- Type I - Crystallographic site eg large grain
- Type II - Porosity
- Type III - Titanium carbide/nitride inclusion.
- Type IV - Aluminium oxide inclusion.

In the case of the EBCHR Alloy APK12 tests, 88% of the samples failed due to Type I sites and 12% failed at Type III inclusions. It is interesting to note, however, that the two Type III failures were both due to single inclusions less than 40 μm big and that all of the Type I sites were smaller than 50 μm in size. There were 22% of samples of P/M Alloy APK 12 which also failed at Type III sites but this time they were all clusters of inclusions which typically increased the total size of the defect to over 100 μm (figure 11). The majority of the rest of the P/M samples (50%) failed at Type II porosity with the remaining 28% fracturing at Type IV oxides. There were no instances of EBCHR failures at either Type II or IV initiation sites. The Type IV oxides found at initiation sites in the P/M samples were all Al rich with traces of Mg and Si and were typically approximately 100 μm in size. The Type II porosity was usually in the region of 100 - 150 μm in size. As well as the size and nature of the defect, its location in the sample is critically important to the LCF life (8). Lives of samples fractured due to surface initiation are generally much shorter than those due to internal initiation. This is certainly shown to be the case in the above tests. In the scatter tests for the P/M material, the five lowest recorded lives were of samples with surface initiated fractures. The internally initiated fracture samples had considerably longer lives, even though in some cases the internal defects were larger than the surface ones. Similarly in the EBCHR material, the two lowest lives were recorded in samples with surface initiated cracks. It was evident however, that the P/M material was more prone to surface initiated failure than the EBCHR material.

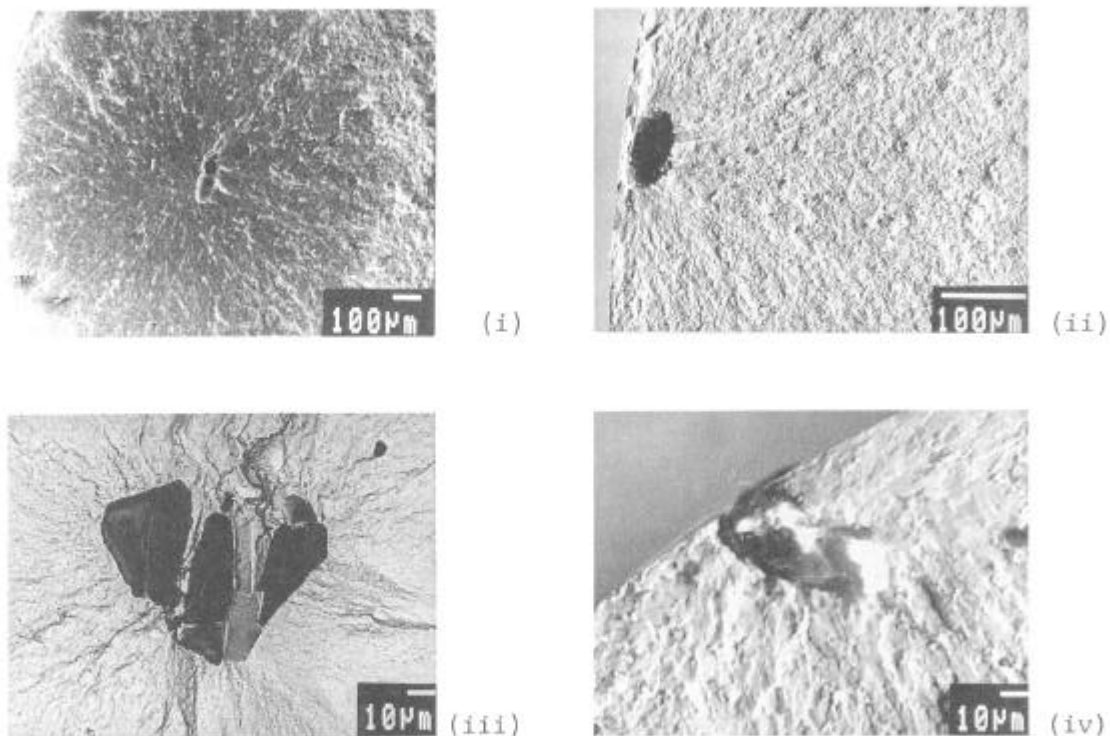


Figure 11 (a) - Fracture initiation sites in P/M LCF samples
 (i) Large internal pore (ii) Surface pore
 (iii) Cluster of Ti nitrides (iv) Large Al oxide defect

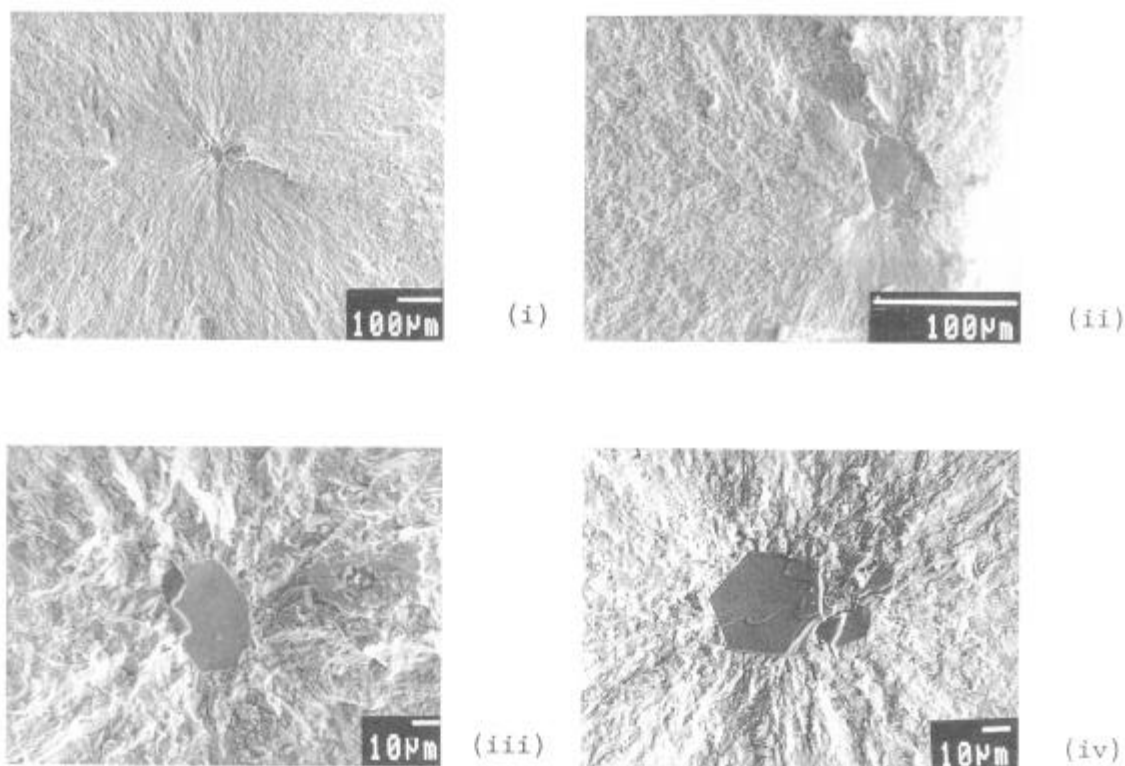


Figure 11 (b) - Fracture initiation sites in EBCHR LCF samples

- (i) Internal crystallographic site (ii) Surface crystallographic site
 (iii) Detail of crystallographic site (iv) Single Ti nitride particle

Discussion

It has been demonstrated that Alloy APK12, which is similar in composition to UDIMET alloy 720, can be successfully melted by Electron Beam Cold Hearth Refining without any problems of segregation and with improved cleanliness compared to -150 µm mesh P/M Alloy APK12. The microstructures obtained in the extruded billet and the final isothermally forged and heat treated discs are fine grained (ASTM 10 - 11) and similar for both types. The tensile and crack propagation results show that the inherent qualities of both versions of Alloy APK12 are comparable and that the EBCHR process has no detrimental effects on these. It is in the critical property of low cycle fatigue life,

which is central to the disc lifing calculations for the alloy, where the benefits of the increase in cleanliness are evident. The results show that as well as obtaining a reduced level of scatter over a dozen samples at one testing condition, the average lives are more than trebled. The fact that the only two (out of 18) EBCHR samples which failed due to anything other than crystallographic features did so at internal titanium nitride particles less than 40 µm in size after lasting for over 150,000 cycles (compared with 9 out of 18 P/M samples failing at ceramic defects up to 100 µm in size) shows that the reduction in number and size of low density inclusions greatly increases LCF lives. It is also clear that if the isolated large grains can be eliminated in the EBCHR material by optimising the TMP conditions, the already reduced scatter band would be narrowed even further. This would increase the "cashable" benefits of the improved cleanliness and LCF property by enabling longer average lives to be built into disc lifing calculations and therefore reducing costs.

Conclusions

- (i) Fine grained (ASTM 10 - 11) forged helicopter discs, comparable in microstructure to P/M material, have been successfully produced from EBCHR Alloy APK12 using cast/wrought techniques.
- (ii) Compared to -150 μm mesh P/M Alloy APK12, the EBCHR material has fewer and smaller (25 μm vs 100 μm) oxide particles.
- (iii) Tensile and crack propagation properties for both variants are comparable.
- (iv) The critical property of low cycle fatigue life, which is greatly dependent upon the number and size of low density inclusions present in a material, was significantly better in the EBCHR Alloy APK 12.
- (v) At 600°C and 1 Hz sinusoidal wave frequency, the EBCHR material exhibited average lives over three times longer than the P/M material.
- (vi) The scatter obtained over twelve samples at one condition was reduced from well over an order of magnitude for the P/M material to significantly less for the EBCHR alloy.

Acknowledgements

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References

- (1) F. Shimizu et al, "Electron Beam Cold Hearth Refining of INCONEL alloy 718 and resulting properties, High Temperature Materials for Power Engineering", 1990, Liege, Belgium, 1675 - 1686.
- (2) S.J. Patel et al, "Electron Beam Cold Hearth Refinement Processing of INCONEL alloy 718 and NIMONIC⁺ alloy PK50", Superalloys 1988, Eds. S. Antolovich et al, Seven Springs, USA, 397 - 406.
- (3) S.J. Patel, "Evaluation of Cold Hearth Refined INCONEL alloy 718", Superalloys 718, 625 and various derivatives, Ed. E. Loria, 1991, Pittsburgh, USA, 43 - 51.
- (4) S.J. Patel and I.C. Elliott, "Cold Hearth Refining and Cleanliness Assessment of Nickel Base Superalloys by EB melting", Electron Beam Melting and Refining State of the Art 1989, Ed. R. Bakish, Reno, USA.
- (5) C.E. Shamblen, D.R. Chang and J.A. Corrado, "Superalloy Melting and Cleanliness Evaluation", Superalloys 1984, Eds. M Gell et al, Seven Springs, USA, 509 - 520.
- (6) H. Pannen et al, "EB Cold Hearth Melting - Process Development for Superalloy Refining", Electron Beam Melting and Refining State of the Art 1990, Ed R. Bakish, Reno, USA, 83 - 97.
- (7) P.N. Quested and S. Chakravorty, "Electron Beam Button Melting as a Cleanliness Assessment Technique for Superalloys", Electron Beam Melting and Refining State of the Art 1989, Ed R. Bakish, Reno, USA, 10 - 35.
- (8) D.R. Chang, D.D. Krueger and R.A. Sprague, " Superalloy Powder Processing, Properties and Turbine Disk Applications, Superalloys 1984, Eds. M. Gell et al, Seven Springs, USA, 245 - 273.