

MSE307 Engineering Alloys 2014-15 L2: Alloy Melting and Forging

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From our previous discussion of defects, it became evident that the minimisation of porosity and ceramic inclusions, and the control of composition, is a key requirement for the manufacture of high performance materials. In fact, for fatigue-limited structures there may be a sense in which the processing defects may control the ultimately attainable properties for many material systems. Therefore we turn now to examine how speciality aerospace materials are produced. For high integrity structures, such as bearing in helicopters, nuclear plant, and so on, great care is taken in the processing to minimise the occurrence of defects such that maximal properties may be obtained.

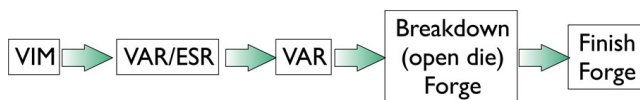


Figure 1: Typical speciality alloy manufacturing sequence for steels or superalloys (simplified).

This manufacturing sequence will, for a high performance steel or nickel alloy, look something like that described in Figure 1. First, the alloying elements are melted together and refined using Vacuum Induction Melting (VIM), followed by Electroslag remelting and vacuum arc remelting. Then, the grain size of the ingot produced is reduced by open die forging to make a billet. This billet is then sliced into sections and the sections closed die forged to final shapes. These shapes are then machined to produce the final parts, and they are joined together.

We will now examine this manufacturing sequence, process by process.

2.1. Melting

The first challenge in making an alloy is to make a homogenous alloy of the correct composition. Often the starting materials will contain excess S, P, O, N etc. and therefore some refining will be required. The advent of vacuum metallurgy in the 1950s-70s enabled transformative changes in this respect, with much lower 'tramp' element contents being achieved. One drawback is that some alloy additions such as Al or Mn, often those with low melting points, will have a low vapour pressure and so will boil off preferentially into the vacuum chamber. This is resolved by limiting the time of exposure of the melt, whilst these elements are present - they are added towards the end of

the melting sequence. Another requirement is to produce ingots that do not suffer from macro-segregation - changes in composition across the ingot. Microsegregation - variations in composition from dendrite core to eutectic at the scale of < 1 mm - can be homogenised away by heat treatment, but macrosegregation cannot and must be avoided.

2.1.1. VIM - Vacuum Induction Melting

A vacuum induction melting (VIM) furnace, Figure 2, costs around US\$3-20 m, and is usually between 3 and 10t capacity. Essentially, it comprises a crucible made of ceramic refractory brick, surrounded by water cooled induction coils, placed in a vacuum. Usually the ingot will be poured from the VIM crucible. When a current is passed through the induction coils, they induce currents in the metal placed in the crucible, and as these are oscillated back and forth, the resistance of the metals in the crucible results in heat generation and hence, melting.

Primarily VIM melting is a refining process - the melt is held in the liquid for several hours, being continuously stirred by the electromagnetic fields. As such, gases dissolved in the melt can be removed, such as P, S, O, N and H. There is very little boiling and some the composition

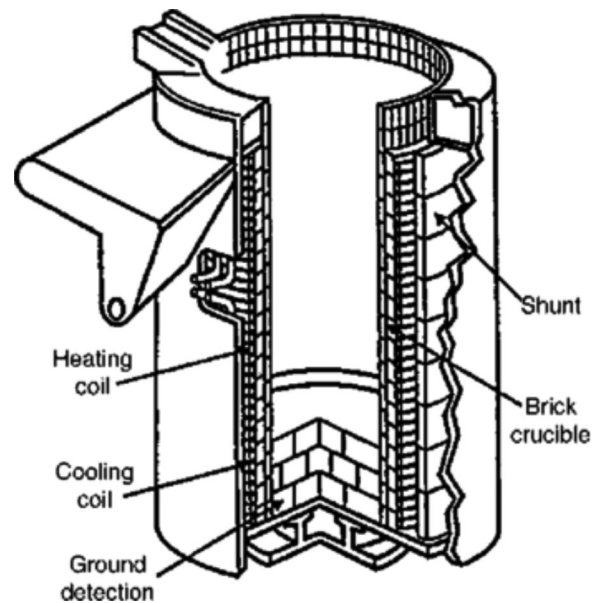


Figure 2: Schematic cutaway diagram of a vacuum induction melting crucible.

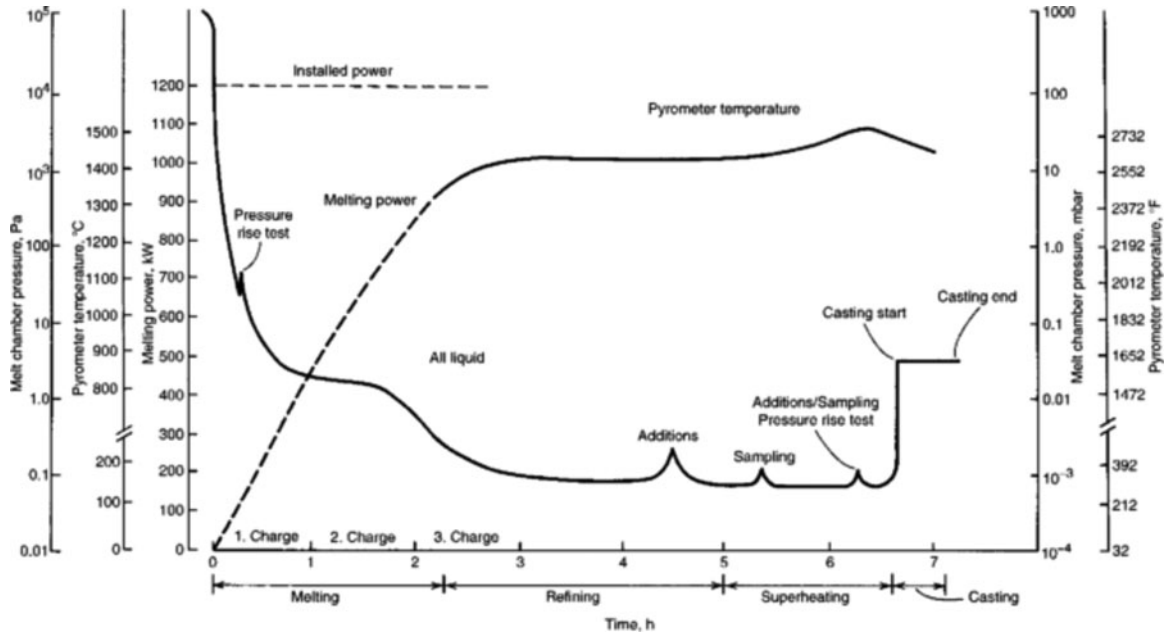


Figure 3: Evolution of process parameters during a vacuum induction melting sequence.

changes slowly. Therefore, it is possible to periodically 'dip' for small samples of the melt, an analysis of which can be used to verify the composition prior to solidification, or to make further additions. And so, very precise composition control is possible.

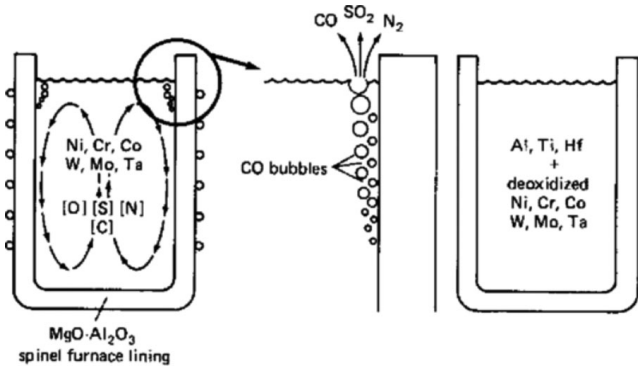


Figure 4: Physical processes occurring during refining in a VIM, and mechanism by which dissolved O is removed.

Over the course of a VIM sequence, Figure 4, first the power is ramped. As the charge heats up, water vapour etc boils off and so the vacuum achieved improves. A quick pause in the vacuum pumps is used to establish that the vacuum system is tight. Eventually, the charge melts and refining begins. Relatively little superheat, ~ 100 K, is used. After a few hours, alloying additions are made and the composition is checked. Once a satisfactory composition is achieved, the chamber to the mould is opened and the crucible is poured. The entire process takes a number of hours.

During melting by VIM, the primary reaction is between C and O, to produce CO, spending dissolved car-

bon to remove O from the melt. The minimum O content is determined by the equilibria with the oxides forming the crucible lining, and so this determines the choice of crucible lining materials. With appropriate materials, O contents as low as 10ppmw can be achieved in Ni superalloys and stainless steels. The CO bubbles nucleate near the top of the crucible, at the interface with the metal, and this can result in mechanical erosion of the crucible, resulting in ceramic inclusions in the melt. As the phrase goes "metals come from ceramics (ores), are melted in ceramics, and are then poured into ceramic moulds - so it isn't a surprise if they contain ceramic inclusions'."

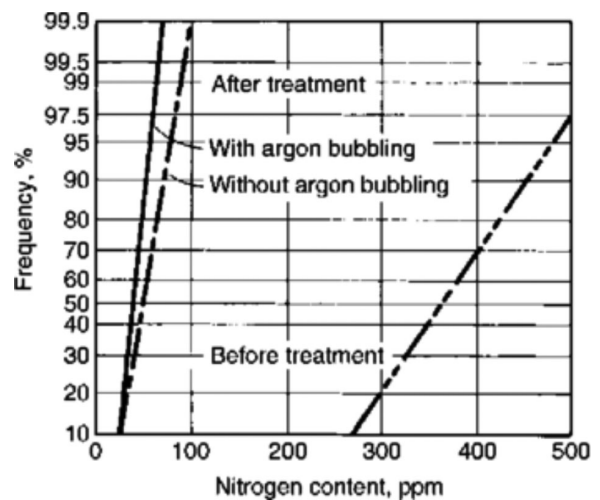


Figure 5: Effect of VIM refining on the frequency of occurrence of melts with a nitrogen content in excess of a specified value, with and without Ar bubbling.

But, VIM is a very effective refining process. Fig-

ure 2.1.1 shows the cumulative frequency of occurrence of melts failing to fall below a given threshold N concentration. Without VIM, 95% of melts contain up to 470 ppmw N, whereas almost all VIM melts contain < 100 ppmw N. This can be further improved by stirring the melt with Ar gas, which allows the continuous exposure of fresh liquid metal to the vacuum without excessive heating by the coils.

Similarly, one can observe that within 2 h of melting, almost all of many of the low melting point volatile ‘tramp’ metallic elements can be eliminated, Figure 2.1.1. There are, of course exceptions like Sn that do not have a high vapour pressure and which must instead be excluded from the feedstock used. But, overall, the introduction of vacuum induction melting resulted in revolutionary improvements in material properties, owing to the elimination of grain boundaries embrittled by, e.g. sulphides and phosphides, and S and P in solution at grain boundaries. Previously, these had been controlled using ‘getter’ elements that precipitated the S and P into precipitates, but they were still undesirable and the getter elements themselves often compromised the properties of the materials. With the introduction of VIM, these elements too could be removed.

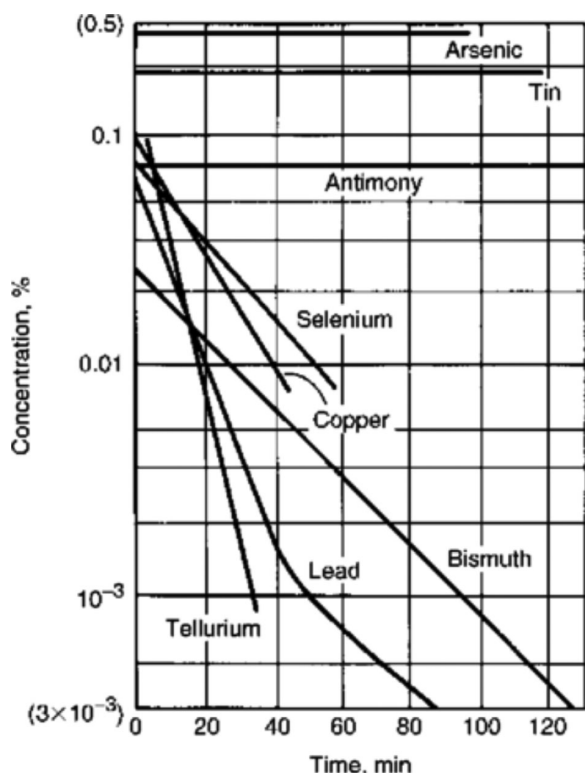


Figure 6: Evolution of the concentration of volatile metals in a VIM melt during VIM refining.

The main problem with VIM melting is the presence of the ceramic inclusions. Many will contend that most subsurface, naturally initiated fatigue cracks in superalloys and steels will be initiated from such an inclusion. A

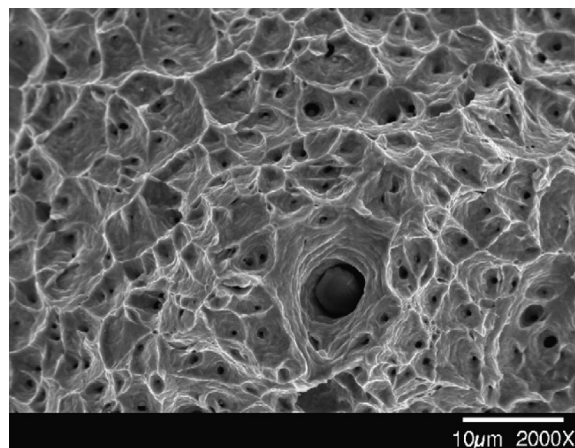


Figure 7: MnS inclusion found nucleating failure in a 316 stainless steel (from San Marchi et al, Int. J. Hydrogen Energy 33(2):889-904, 2008).

particularly pretty example is shown in Figure 7, which happens to be a manganese sulphide (presumably in non-VIM material). Notice that microvoid coalescence has occurred around the initiating defect - the failure was ductile apart from at that inclusion.

2.1.2. VAR - Vacuum Arc Remelting

If VIM is a refining process, then VAR is a solidification process, that is, its purpose is to avoid macrosegregation and produce an improved solidification microstructure. The process works by taking a pre-existing ingot from the VIM, welding it onto a current conductor and then applying a bias voltage (~ 30 V). Essentially, it is like a very big MIG/GMAW setup, with a DC electrode negative setup.

A water cooled Cu crucible is used, so the process is very clean, with no ceramic present. The currents are very high - around 40 kA (it is an O!MW process), and the melt rate is around 500 kg/h - so it takes around 16 h to melt an entire 8 t electrode. The bias voltage creates an arc between the melting ingot and the melt pool, with the arc formed by metal ions evaporated from the pool and ingot. This arc melts drops from the ingot, which fall down into the pool.

As the solidified ingot cools, it shrinks away from the Cu crucible wall, limiting heat transfer. Only the top of the solidifying ingot will be liquid, as shown in Figure 10. As melting proceeds, the electrode melts back and the ingot grows, maintaining the arc gap. Electrode weighing devices are also used for control and monitoring purposes. The process can be modelled, at least to an extent. The melt pool shape largely reflects the isotherms produced due to heat removal to the crucible, but within the pool several effects are at play. The arc buoyancy driven flows due to the temperature variation in the pool, which is heated from the top, as well as electromagnetic stirring from the arc itself. If these currents become too violent, they can break off the dendrite tips from the solidifying material, which (if they don't melt) can then act to nu-

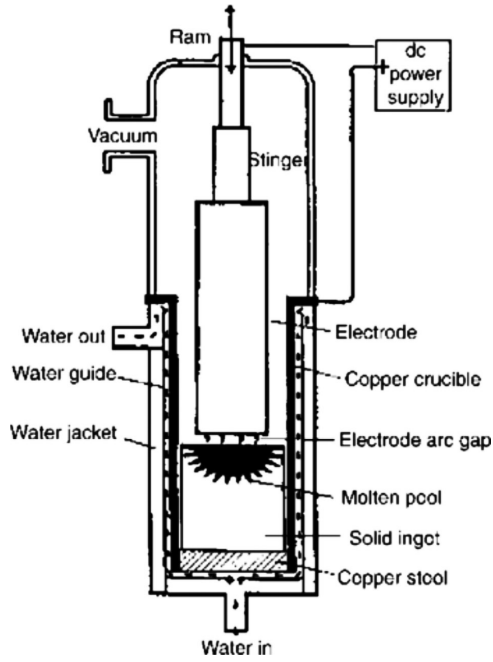


Figure 8: Schematic showing the general arrangement of a vacuum arc remelting furnace.

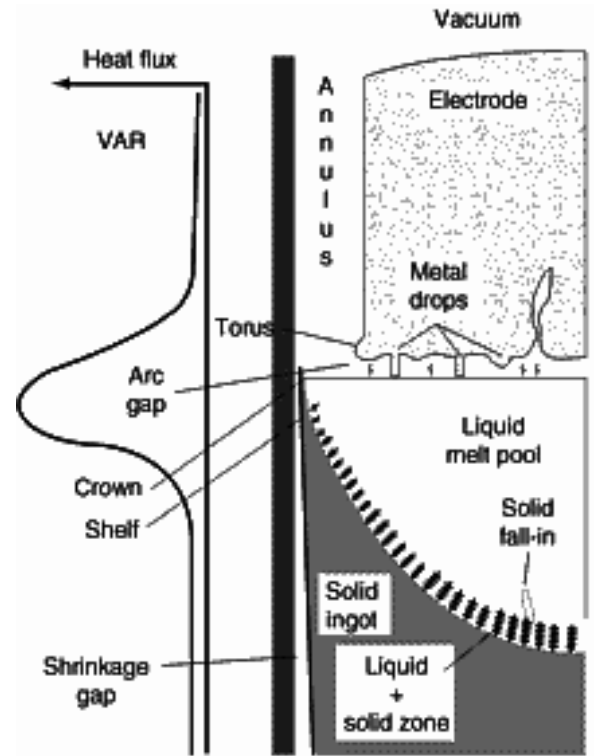


Figure 10: Physical processes and heat flow in the region near the arc in a VAR.



Figure 9: Photograph of the top of a (newly installed) VAR furnace.

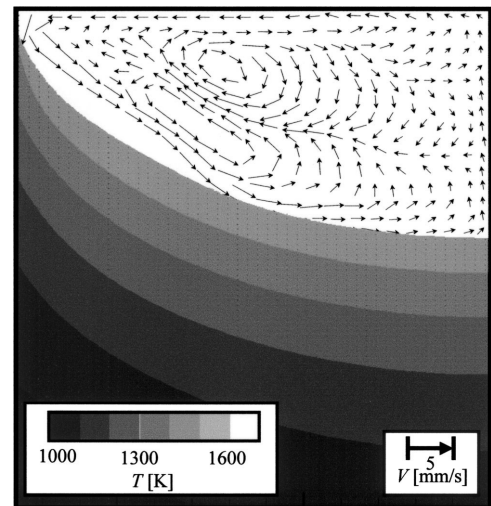


Figure 11: (Predicted) fluid flows and temperature field in an IN718 VAR melt pool, from Wu et al., Metall. Mater. Trans. A 33(6):1805-1815, 2002.

create equiaxed grains. Melting interruptions can have a similar effect, Figure 12, and in extreme cases this can give rise to a type of defect known as a ‘tree ring’ defect, which is manifest as the appearance of rings in macro etched transverse sections of the casting that resemble tree rings.

The primary benefit of VAR melting (in Ni) is that there is directional solidification from the bottom to the

top of the ingot; the melt pool is small and carries little superheat. Therefore the rates of solidification and melting are balanced, and there is no opportunity for macro segregation, which as previously stated cannot be mitigated by heat treatment.

In addition, there is relatively little stirring (compared to VIM) and so oxide inclusions *may* float to the top of

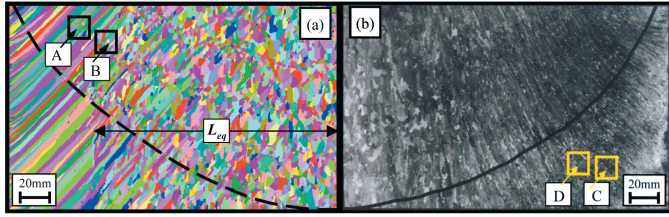


Figure 12: Predicted (left) and observed (right) microstructures in an IN718 VAR ingot subjected to current instabilities during melting, from Wu et al., Metall. Mater. Trans. A 33(6):1805-1815, 2002. The melt pool shape is shown by the black line.

the ingot. This, together with the change in solidification structure at the top of the ingot, means that the top of the ingot is usually discarded.

Finally, the remelting process gives further opportunity for undesirable trace elements to boil off.

2.1.3. ESR - Electroslag Remelting / Refining

Electroslag remelting or refining was developed (in the UK) in response to innovations in the soviet union. The process is similar to VAR, but the current is passed through a protective molten salt slag that floats on the surface of the melt. Typically, the slag is a ternary+ mixture of CaF_2 , CaO and Al_2O_3 , optimised to give the desired melting point (just above that of the alloy) and conductivity. The slag conducts the 'arc', smoothing out the heat flow, as well as protecting the metal from the atmosphere. It is a refining process, because the slag removes oxygen from the melt that drops through it from the electrode and also tends to trap any inclusions. As such, it is a very effective method for 'cleaning' up the inclusion content from VIM.

As such, when it is desired to achieve the very minimum possible inclusion rates, a VIM-VAR-ESR melt route is sometimes used. A consideration of the amounts of energy involved should provide an appreciation that this isn't a cheap way to produce material!

2.1.4. Melting of Ti and Zr

Titanium and zirconium melting is rather special. They are both very reactive metals and so it isn't possible to melt them in ceramic crucibles for hours at a time. As we shall see in the titanium lectures, titanium largely derives its good specific fatigue strength from its lack in intrinsic ceramic inclusions from processing. This is because liquid titanium can dissolve its own oxide, and so it really is possible to produce inclusion-free titanium ingot. Therefore both Ti and Zr are melted by so-called triple VAR ($3 \times \text{VAR}$) processing. Sponge from the Ti production process is compacted to form an electrode, together with alloying elements, and this is then used directly in the VAR process.

However, as shown in Figure 2.1.4, titanium is a poor conductor of heat and so the solidification part of the process is quite different. At the end of melting, as the current is ramped down during so-called 'hot topping,' then melt

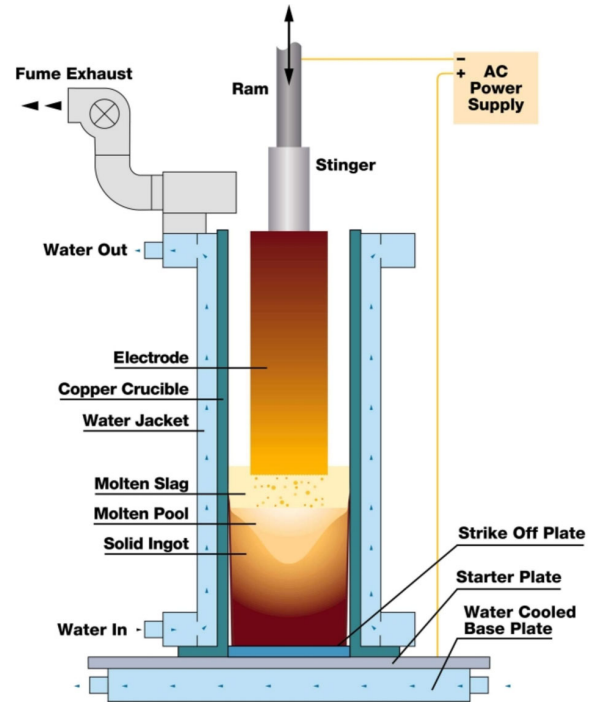


Figure 13: Schematic showing the general arrangement of an electroslag remelting / refining furnace.



Figure 14: Photograph of the top of an ESR furnace.

pool is still very deep and most of the ingot is molten. There is still very little superheat, though. At this point, nucleation of new solid happens at the top of the molten pool, but the solidifying crystals are more dense and therefore 'rain' down into the solid. Therefore a columnar-to-equiaxed transition is observed in the ingot.

In addition, as the process has very little superheat,

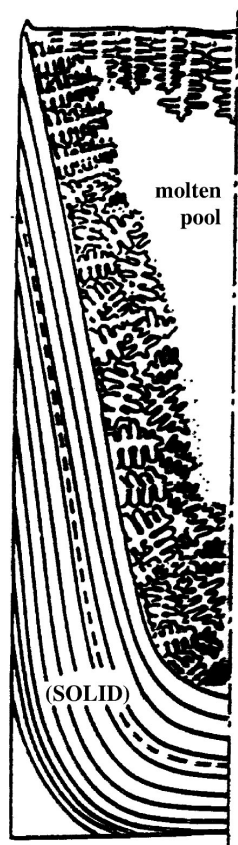


Figure 15: Schematic of the microstructure formation and isotherms in a titanium VAR ingot at the end of melting / beginning of hot-topping. From A. Mitchell, Mater. Sci. Eng. A 413-414:10-18, 2005.

any large chunks of high melting point refractory metal present in the electrode can drop straight through the melt without significant dissolution occurring. Therefore problems can sometimes occur with the use of inappropriately sized additions of refractory metals like Mo. In particu-

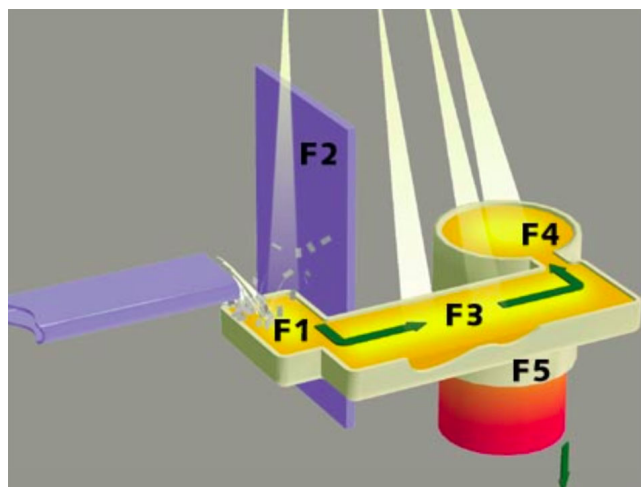


Figure 16: Schematic of the Electron Beam Cold Hearth Refining (EBCHR) process.

lar, W is banned from titanium plants, most notably in ball point pens. Such inclusions are called 'high density inclusions' or LDI where they occur, although it should be emphasised that in well controlled plant the occurrence of HDI is very rare.

The other process problem in the VAR melting of Ti can be the occurrence of high oxygen inclusions (low density inclusions or LDI). These are typically high O/N chunks of titanium up to a few mm in size, that do not dissolve during the melting process. Typically they are inherited from the Ti reduction (Kroll) process, from the parent Mg feedstock. Improvements in the water and vacuum systems in Kroll and Mg plant in the 1960s-90s have more-or-less eliminated the occurrence of LDI.

However, these issues have meant that traditionally, it has not been possible to recycle Ti chips from machining. This is because WC chips from cutting tools, and overheated O-contaminated chips could result in HDI and even LDI. Therefore all rotor-grade material, and most aerospace material used is rotor grade, has to be made from virgin sponge. As the majority of the forged material is machined away, this represents a problem. 70% of Ti produced is used in aerospace, so the non-aero market is limited, and much lower value - mostly chemical plant and armour, which are both cost sensitive. Historically, much chip ended up as additions to steels.

Recently, however, a new process has emerged called Electron Beam Cold Hearth Refining (EBCHR). Here, electron beams are used to melt a keep liquid of molten pool of titanium contained in a water cooled Cu crucible. The crucible is cold and so a solid Ti 'skull', of the composition of the alloy, is produced and the molten Ti is only ever in contact with Ti. At intervals, the melt is tapped and poured; the ingot produced is then fed into VAR. The advantage is that the residence time in the melt is high enough to dissolve any LDI, and any HDI simply drop into the skull. There is enhanced Al evaporation that needs to be managed (by adding more Al), however. This process has meant that some manufacturers can now recycle Ti chip, free from the fear of embrittling inclusions.

2.2. Forging

Cast ingots are first skim machined to remove any oxidation from the surface, and then must be hot worked. They will first be homogenised in a furnace to homogenise the composition variations arising due to micro segregation in casting. The purpose of initial hot working is to reduce the grain size from the cm-mm to the hundreds of μm . This needs to be done in a single phase field, where lots of slip systems are available and deformation can occurring homogeneously, such that large strains can be sustained without cracking. Once the grain size has reduced such that more homogenous deformation can occur naturally, then the temperature can be lowered.

Therefore the initial forging to reduce the grain size is performed at high temperatures. High strain rates are used with open-die forging. Each 'bite' by the forging press



Figure 17: Rough machined VAR ingots on a factory floor, awaiting forging.

produces local deformation around the bit, at which point the ingot is moved and another bite is taken. The plastic strain from each bite also results in local adiabatic heating, which (combined with the large size of the ingot) means that the temperature is kept high enough to work the ingot for many minutes before reheating in the furnace. Such open die forging is also called ‘cogging.’

During each ‘hit’, dynamic recrystallisation can take place, and also static recrystallisation can occur after deformation whilst other parts of the billet are being worked (in forging, the piece is called a billet - in casting, an ingot). Figure 19 shows this effect (the scale is in ASTM grain size, where low numbers indicate large grain sizes - ASTM no. 1 is around 0.3mm diameter, no. 5 is 60 μm and no. 10, 11 μm .). If the cogging process is performed incorrectly (small, fast bites), then deformation doesn’t reach the centre of the billet and localises at the surface, whereas if it is done well, then a relatively even grain size of < 100 μm can be produced. “Dead zones” underneath the flats of the dies must also be managed.

Cogging presses are relatively small - around 5000 t would be typical. For cogging to be performed, therefore, there must be a temperature interval in which the alloy is below its melting point but in a single phase field. This must be fairly wide, 50 – 100 K, in order to allow for adiabatic heating and for cooling of the part during cogging.

After cogging, the billet is then rolled to plate, or cut



Figure 18: Photograph of a billet being open die forged (cogged).

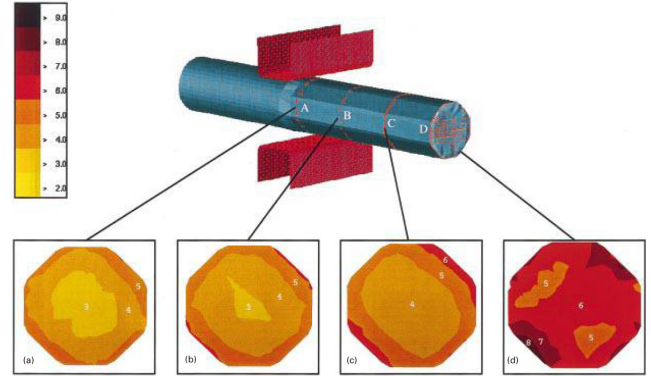


Figure 19: Finite element simulation of the cogging process, showing the evolution in grain size along the length of the billet. From Dandrea et al, Superalloys 2000, pp85-94.

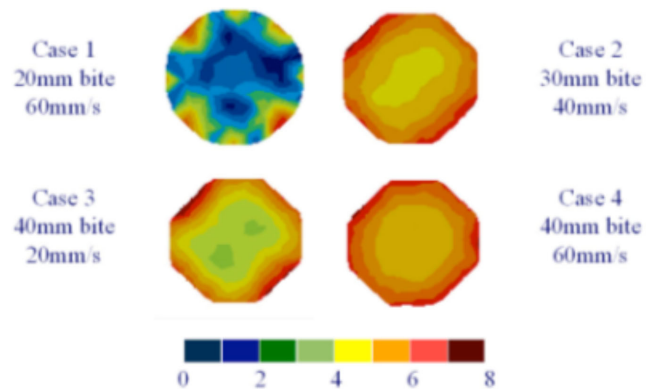


Figure 20: Effect of bite sizes and speeds on the grain sizes obtained in cogging, according to a finite element simulation. From Dandrea et al, Superalloys 2000, pp85-94.

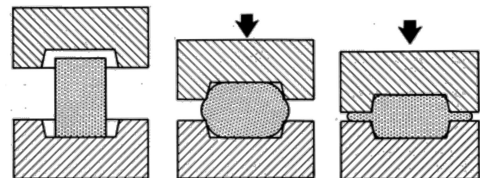


Figure 21: Schematic of the closed die forging process, illustrating the metal flow.

into slices which can then be closed die forged. Closed die forging is usually performed in air using tool steel dies. This compresses a cylindrical work piece into a rough shape, Figure 22. This rough shape is then machined to an approximation to the final component shape. This can then be ultrasonically inspected and macro etched. Final machining then proceeds and any finishing operations like shot peening. The plasticity associated with shot peening tends to contraindicated ultrasonic non-destructive inspection (NDI) and therefore NDI is performed prior to the finishing operations.

Closed die forging will sometimes be performed on material that is in the two phase field, so as to avoid excessive strain localisation, although this depends on the alloy.



Figure 22: Photograph of a turbine disc forging, in IN718. Notice that the forging blank is much larger than the final disc shape - often > 90% of the material is machined away.

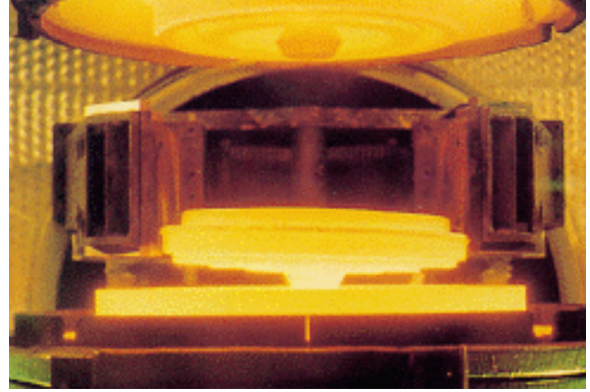


Figure 24: View into an isothermal forging press after opening of the Mo dies to view the P/M Ni superalloy part.



Figure 23: The 75,000 tonne (750 MN) press at VSMPO in Verkhanya Salda, Russia. The component is an 8-ton B-777 Ti-10-2-3 truck beam forging, with a man for scale.

But, conventionally using tool steel dies then for forging temperatures of 800 – 100 °C for Ti and Ni alloys, it must be performed quickly, because the temperature capability of the tool steels is only around 500 °C, and so excessive heating of the dies cannot be tolerated. Therefore the strain rates can be large, > 0.1 s⁻¹.

Figure 23 shows the 75,000 t press at VSMPO used to make the large truck beam forgings for the 777 and 787. This press has a higher load capacity than any in North America, and is forges entire 8-tonne billets at once.

As an aside, it is worth mentioning isothermal forging. Latest-generation Ni disc alloys are made by hot isostatic pressing of alloy powders, typically produced using inert gas atomisation from the VIM. These powders are quite fine grained (~ 10 μm). The powder compact is then forged, and possibly extruded. The final forging step is isothermal forging. These alloys do not possess a wide enough single phase region for conventional closed die forging and so forging is performed slowly, at constant

(iso) temperature - strain rates of 10⁻² might be normative. As the strain rates are low, the dies must be heated to avoid chilling the part, and so Mo dies have to be used (TZM). Unfortunately, Mo suffers from rapid oxidation at these temperatures, and MoO is itself volatile, and so the entire operation must be performed under vacuum. Thus, isothermal forging is very expensive. It does, however, result in premium fine grained product, with exceptional fatigue performance.

Once the part has been forged, it is then aged, machined, inspected, any surface treatments are performed and then finally it is joined to its neighbouring components into an assembly. In terms of joint efficiency - the efficient transfer of load - the continuous joint provided by welding is desired. Unfortunately, this then results in a relatively uncontrolled microstructure in the weld region and adjacent heat affected zone. Final heat treatments are often then performed to manage the weld stresses, as well as optimise the precipitate distribution, as well as any final surface treatment at that stage.

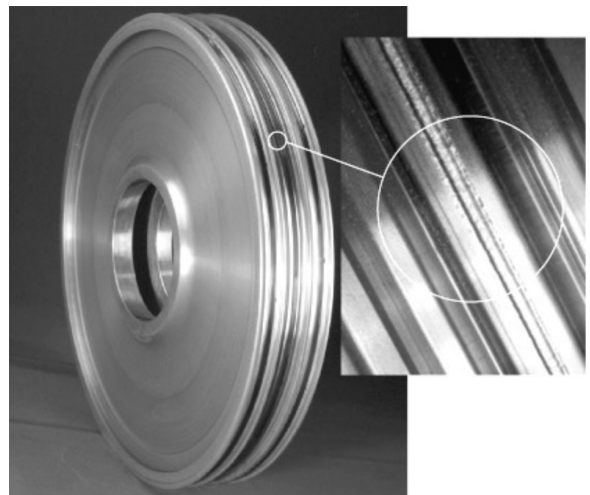


Figure 25: Electron beam welded assembly of two Ni superalloy discs. Inset shows the profile of the electron beam weld.