

OPTIMIZATION OF MATERIALS PROPERTIES

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Summary

The optimization of microstructure in structural engineering materials through processing is an essential factor in achieving satisfactory performance in service. The development of microstructure through solidification, mechanical processing and heat treatment, are considered in turn. The structural defects, which can occur, are examined and interactions between the process steps are considered. In each case, practical examples are presented to illustrate the principles involved.

1. Introduction

There are two principal requirements that have to be achieved in the processing of materials for engineering applications. First, the material must be shaped into the form required for the component being produced. Second, it must, at the end of processing, possess an internal microstructure, which can provide the mechanical and physical properties required for the service application of the component. Only in a very limited number of cases can these two requirements be considered or dealt with separately. The processes used to engineer shape also affect microstructural development, either directly

or indirectly, by changing the response of the material to further thermal treatments designed to alter microstructure. The following sections discuss the principal methods by which microstructure and shape can be achieved. The division into sections should not, however, be considered rigidly, since in many cases, several of the methods may be employed in order to achieve an optimized microstructure.

2. Optimization through Solidification Processing

Historically, the requirement to produce a particular shape was the dominant factor and microstructure was compromised in order to achieve this. Casting is a process developed as one of the earliest processes for making metallic implements. It has the strong advantage of producing a net, or near net, shape. In terms of the process the mold or die design must be such that complete filling with metal can be achieved without trapping air within it, requiring good design of feeders and risers. However, metallurgically casting has some inherent drawbacks. First, the solubility of gases generally decreases sharply with the transition from liquid to solid (notably hydrogen in aluminum) and the evolved gas, if trapped, will produce gas porosity in the final casting. Degassing prior to casting is required to mitigate this phenomenon. Second, engineering casting alloys all shrink on solidification, a phenomenon which can result in the formation of shrinkage voids and internal stresses in the casting. Again the design of the mold to facilitate the access of fresh liquid to regions of solidification shrinkage can help to reduce the number and size of voids and choice of casting alloy (changing melt fluidity and solidification temperature range) can be exploited to further improve the situation. Third, turbulent flow taking place in mold filling can result in the entrapment and folding over of surface oxide skin into the final casting, with serious consequences for casting strength and toughness. Beyond this, the mechanism of solidification in metals and alloys is itself, of very considerable importance in determining final casting properties. Solidification will commence with relatively little undercooling and typically begins on the mold walls. Nucleation sites are plentiful and a very fine equiaxed grain microstructure (the chill zone) is formed unless the melt superheat was so great that the mold itself becomes heated to a temperature above the bulk melt liquidus temperature (an unlikely scenario). Growth of these crystals will be controlled, both in rate and direction, by the heat flow. It will be fastest anti-parallel to the directions of fastest heat loss from the system (in a simple shape growth will be at 90° to the mold walls). Since the growth of crystals is crystallographically anisotropic, those grains whose fastest crystallographic growth axes are closest to parallel to the thermally preferred direction of solid growth will undergo faster development than others and they will form long columnar crystals which will exhibit strong crystallographic texture. This texture will, in itself, modify the mechanical behavior of the casting and render it locally anisotropic. More seriously, where two such growth fronts meet the interface will be high angle and is likely to contain insoluble inclusions originally present in the melt or picked up from the mold and any products of invariant reactions which the melt may undergo on solidification (a function of alloy chemistry). Such interfaces represent planes of mechanical weakness within the casting and, in extreme cases, cracking can occur under the action of shrinkage stresses post-solidification. Careful casting design and control of heat transfer (e.g., by use of chill plates to stimulate local solidification) can be employed to reduce these factors. Finally, in alloys and under appropriate thermal conditions, a central zone of equiaxed crystals can be produced as a consequence of

constitutional supercooling. The casting can, therefore, exhibit microstructurally diverse regions with varying mechanical properties. The desirability of eliminating this variability and producing a generally equiaxed microstructure underlies the important industrial development of additives which act as inoculants to stimulate grain nucleation and produce a refined and more equiaxed microstructure. In the case of alloys, in which there is no fixed melting point, but rather a melting range of temperature (between liquidus and solidus), it is possible to carry out casting of the alloy in this range, where both liquid and solid co-exist. This has several advantages: first, the solid can consist of small particles, leading to a fine grain size post-solidification, and second, there is much less contraction on complete solidification, reducing shrinkage cavity formation and residual stresses. The price paid is the much less fluid nature of material, which decreases with increasing volume fraction of solid. This all assumes that the castings are to be employed at low homologous temperatures where a fine and equiaxed grain structure is generally beneficial in terms of mechanical properties exhibited by the casting. However, at high homologous temperatures, time dependent deformation under stress (creep) is an important mode of failure and one of the principal mechanisms for this process is the diffusionaly accommodated sliding of grains past one another along their grain boundaries. In such cases there is benefit to be obtained from enlarging the grains or even eliminating them altogether. This has been achieved very successfully in high temperature nickel-based superalloys employed as cast turbine blades for gas turbine engines (see *Aerospace and Space Materials*). The major stress axis in this case is parallel to the blade length. The use of a chill plate and unidirectional heat flow can be exploited to ensure that the metal solidifies in a columnar manner in which the grain boundaries are predominantly aligned and contain the stress axis. Such boundaries do not experience a driving force for sliding and creep strength is thereby enhanced. This process has been further developed such that, after solidification begins on the chill plate, the crystals have to grow through a growth selector, a region of three dimensional geometry designed to progressively cut off the growth paths of all but one of the grains (e.g., a spiral section consisting of one or more complete turns) such that on emerging from this region, the solidification front consists of a single crystal, which then forms the body of the blade. Although simple in principle, the success for the process depends critically upon a combination of mold design, thermal control and alloy chemistry all of which have to be matched to each other.

The use of pressure, exerted on the liquid, during mold (or die) filling is employed industrially to ensure complete filling and production of complex shapes with replication of fine surface detail. A similar principle is employed in the production of polymeric and glass products via solidification processing, the simplest examples being bottles, which are made using gas pressure to press the material against the die: in this case the high viscosity of the liquid is exploited, so the process is not applicable to liquid metals which have much lower viscosity. Since both the polymer and glass are typically non-crystalline, there is not the same concern with microstructure development in processing, which is associated with solidification casting of metals.

Pressure is also employed in the production of composite materials by infiltration of a liquid matrix phase into a preform of another material, typically in the form of long or short fibers. Examples include polymer resin infiltration of carbon fiber preforms (resin transfer molding) and liquid metal infiltration of ceramic preforms to produce metal

matrix composites (MMCs). The preform consists of the material to be infiltrated, either woven in a mat or held together by use of a binder to provide a network of material with interspaces between the individual fiber segments into which liquid can be infiltrated. In the case of MMCs, the ceramic is generally not wetted by the ceramic, and pressure is required to overcome the capillary resistance presented in the interspaces between the fibers, as well as the frictional resistance to liquid flow between the fibers (itself a function of melt viscosity). For polymer composites, the situation is a little easier as the fibers are generally wetted by the resin. The complexity of the local fluid flow into the preform makes gas entrapment and the occurrence of non-infiltration defects more likely than in conventional casting but the scale of the defects is generally smaller. The volume fraction and orientation of the fibers is employed to locally modify the material properties, both mechanical (e.g., stiffness, strength and wear resistance) and physical (e.g., density and conductivity). Components can be produced that are either fully reinforced or contain the fibers in selected locations, such that the properties are tailored to the local engineering requirements. For example, diesel engine pistons can be manufactured in aluminum alloy by die casting. The crown of the piston can be locally reinforced with ceramic by use of a shaped short fiber preform (typically alumina based). The MMC so produced has improved wear resistance and thermal fatigue resistance, which is needed in the material exposed to the high temperatures resulting from fuel combustion. The cooler regions in the body of the piston exploit the greater toughness associated with the unreinforced matrix alloy. Composites containing particulates can also be made by liquid infiltration of preforms or, more simply, by stirring particles into the liquid prior to casting. In the case of metal matrix composites, the solidification structure produced within the composite is modified by the presence of the ceramic, which can inhibit crystal growth. If the ceramic is not held in place in a preform, it can undergo pushing by the melt, as a consequence of the non-wetting behavior, and become concentrated between the solidified alloy grains. Chemical reaction between the melt and ceramic may also occur, the nature and severity of this being dependent upon the chemical nature of the matrix alloy and the ceramic and the thermal conditions employed in casting. In general, such reactions are highly deleterious to the development of required properties in the composite and preclude the use of highly reactive liquid metals, such as titanium. However, there are materials that actually exploit chemical reactions in the melt to produce a composite. The melt acts as a solvent into which reactants are dissolved. A chemical reaction takes place producing a product, which is precipitated from the melt to form a particulate reinforcement in the subsequently solidified metal. The size and distribution of the reinforcement can be modified by close control of the process conditions. The reinforcement is necessarily in equilibrium with the matrix material, and has atomically clean interfaces with the matrix, resulting in good interfacial bond strength, which may be lacking in other MMCs where some degree of interfacial reaction can occur. Titanium and titanium aluminide alloys containing dispersions of titanium boride particles can be produced with refined grain size and improved mechanical properties by this route.

3. Optimization through Mechanical Processing

The ability to modify microstructure through mechanical processing is extremely limited for non-metallic materials, which are generally brittle at low homologous temperatures and undergo viscous flow in the glassy state at high homologous

temperatures. Engineering alloys, however, possess the ability to undergo permanent or (plastic) deformation even at low homologous temperatures via the motion of dislocations and, in some cases, by mechanical twinning or martensitic transformation of crystal structure under an applied load. At low homologous temperatures, interactions between dislocations lead to the creation of dislocation locks and tangles, which impede further motion and which necessitate an increased applied stress to continue deformation as the dislocation density within the metal increases. This phenomenon of work hardening is exploited in metals deformed at low homologous temperatures (cold worked) to increase mechanical strength, but at the price of reduced ductility. As temperature is increased, the material can undergo recovery (involving cross-slip and climb of dislocations past obstacles) or recrystallization (the nucleation and growth of undeformed crystals which consume the deformed structure). The rate of work hardening diminishes and can fall to zero. While this makes permanent, or plastic, deformation easier, the lack of work hardening renders the material unstable under tensile loading in which the material extends parallel to the stress axis and contracts normal to it. Any small locally larger reduction in the dimensions of an initially uniform load bearing section will, in the absence of work hardening, become unstable against further deformation and tensile failure will occur at this site without additional extension of the bulk of the material, a process known as necking. For this reason, all mechanical working processes carried out at high homologous temperatures (e.g., forging, rolling, extrusion) avoid the imposition of tensile forces. Only at low homologous temperatures where work hardening can stabilize the section against necking, can tensile forces be employed to shape a product, most notably in wire drawing (see *Materials Processing and Manufacturing Technology*).

Common engineering alloys are produced as large cast billets and must undergo substantial mechanical deformation to convert the billet to a usable shape (e.g., plate, sheet or rod): this can only be performed in the absence of work hardening at high homologous temperatures. The processing breaks down the as-cast structure, and produces a new grain structure. For most applications, a fine grain size is desirable, as it gives improved combinations of strength, toughness and ductility and thus, the hot working schedules must be carefully designed to produce the desired shape change in as economical a manner as possible while also fitting the desired grain structure. For example, control of the microstructure and composition of low carbon structural steels for structural applications in civil, marine and chemical engineering, where large quantities are used, typically in simple product forms (plates, pipes) and typically in sections some millimeters in thickness. Strength and weldability are required service properties, and are achieved by keeping the carbon content of the steel low to improve the latter, while refining the grain size to increase the former. Reduction in grain size is achieved by controlled rolling. The key is to create as large a grain boundary area in the deformed austenite (face centered cubic (fcc) high temperature phase in Fe-C alloys) upon which ferrite (body centered cubic (bcc) phase in Fe-C alloys stable at lower temperatures) grains can nucleate during cooling, discouraging recrystallization and grain growth in the austenite, while encouraging nucleation of the ferrite and inhibiting grain growth in that phase. To this end, the steel is alloyed with very small amounts of transition elements (vanadium, titanium and niobium) which form carbo-nitride particles at about 1150 °C, and inhibit growth of the austenite grains. The metal is rolled in the temperature range where the austenite recrystallizes during deformation (~1150–

1050 °C) allowing working loads to be kept low. It is finish rolled at a lower temperature (~900–800 °C) at which the austenite only undergoes recovery and the austenite grains become flattened and have a large surface area per unit volume. These grain surfaces act as nucleation sites for the ferrite grains, which form on further cooling: the larger the number of nucleation sites per unit volume, the finer the final grain size. Careful control of rolling schedule results in a microstructure consisting of an ultrafine grain (2–10 µm) ferrite of low carbon content which is further strengthened by additional precipitation of alloy carbides which occurs when austenite transforms to ferrite. This combination of grain refinement and alloy carbide precipitation results in very high strength in the hot rolled condition, while retaining ductility and enhancing toughness. Where a high surface finish is required in the product, cold rolling is employed. This necessarily work-hardens the material, increasing its strength but decreasing the ductility. This can be acceptable for many applications including relatively mundane products such as aluminum kitchen foil, but in cases where extensive forming is required to achieve the final shape, annealing (heat treatment at an elevated temperature to cause softening by recovery or recrystallisation) may be required. The hardening resulting from cold work is also exploited in some engineering components to effect local hardening in order to improve service behavior, or to repair defects introduced at an earlier stage in processing, or as a consequence of service exposure. Shot peening is used to harden surfaces and place them in compression to render them more resistant to fatigue cracking. Hot isostatic pressing is also employed to seal internal voids in engineering components including titanium alloy castings and creep damaged turbine blades.

Formability rather than strength is the key property required for many applications involving the use of thin sheet. This depends not only upon the shape and size of grains within the metal, but also upon the range of crystallographic orientations they display. A single crystal of a metal is necessarily anisotropic in its properties, elastic and plastic, but in a polycrystalline metal in which grain orientations are random, this anisotropy is lost. However, if grain orientations are not random (the material possesses a crystallographic texture) a degree of anisotropy will be re-established. Texture can be introduced by directional solidification, as noted earlier, and by deformation and recrystallization of metals. In the case of aluminum-magnesium based alloys used for the production of beverage cans, control of texture in the rolled canstock is essential to the formability by deep drawing. The presence of texture can result in “earing”: non-uniform deformation in drawing, which results in the top edge of the can body having a wavy profile necessitating trimming off material to produce a straight edge. To avoid this, the alloy ingot is homogenized and slowly cooled after homogenization (a high temperature soak for a sufficient period for compositional inhomogeneities introduced during solidification to be eliminated by solid state diffusion), to about 510 °C, to produce a fine uniform manganese based dispersoid, which provides good control of grain size on later recrystallization. It then undergoes a sequence of hot rolling reductions and annealing treatments to develop a mix of deformation and recrystallization textures in the finished thin sheet material. The internal microstructure and texture are, thereby, optimized for the process of deep drawing and wall ironing used to form the can shape, while inhibiting earing.

In addition to exploiting hot deformation and annealing to modify texture, it is also used

to control grain size, shape and phase distribution to optimize the material for further shape processing. Notably, alloys such as titanium 6% aluminum 4% vanadium, can be thermomechanically processed to produce sheet with a very fine duplex grain structure of α and β grains a few microns in diameter which, at ~ 930 °C, co-exist in approximately equal volume fractions. Such a microstructure can be further hot worked into complex panel structures by superplastic forming (SPF). Superplastic formability can also be achieved in a range of alloys, including steel and aluminum, and even ceramics by developing specific microstructures through controlled processing.

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Biographical Sketch

Harvey Flower is a graduate of Cambridge University and presently Professor of Materials Science at Imperial College of Science, Technology and Medicine in the University of London. His research interests center on electron microscopy and light alloy development with emphasis on aerospace applications. He was awarded the Imperial College Armstrong medal in 1974 and the Rosenhain medal of the Institute of Materials in 1988 for his research on titanium and aluminum based alloys. In recent years his research has extended into metal matrix composites with particular emphasis on low cost net shape cast aluminum based materials. He is author, or co-author, of about 170 scientific papers and has edited

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