THE HALL-PETCH RELATIONSHIP

Introduction

One of the underlying principles in materials science is that properties can be deduced from a knowledge of the microstructure. By microstructure we mean the crystalline structure and all imperfections, including their size, shape, orientation, composition, spatial distribution, etc. A list of the types of imperfections would include point defects (vacancies, interstitial and substitutional solutes and impurities), line defects (edge and screw dislocations) and planar defects (stacking faults, grain boundaries), second phase particles and dispersoids or relatively large amounts of other phases. With so many factors to consider it would seem that finding this structure-properties relationship would be impossible. But selecting the type of material to use in the study (pure/alloy, single crystal/polycrystalline, single/multi-phase) one can eliminate many factors and concentrate on the ones of interest. In this experiment a single-phase alloy is used in the investigation of the influence of grain size on mechanical properties. It will concentrate on the relationship between grain size and yield strength, the Hall-Petch relationship, but it will also consider its effect on other mechanical properties.

Background

Studies of the mechanical properties of single crystals have taught us that many of their fundamental properties can be attributed to the type of crystalline lattice. For example, fcc metals slip at stresses as low as a few hundred psi but they also rapidly strain harden. This is due to the close packed nature of the fcc lattice and its 12 slip systems. This large number of slip systems makes it practically impossible that a slip plane will not be oriented so that the critical resolved shear stress is low. However, it also means that slip will be likely to occur simultaneously on different slip planes, leading to rapid strain hardening. The significance of findings like these is easier to appreciate when one compares them to the behavior of bcc materials, which require much higher stresses to initiate slip, and hcp materials, which have a very limited number of slip systems, and to polycrystalline materials, which adds another whole level of issues to consider.

Although single crystal materials are occasionally used as structural materials, polycrystalline materials are by far more commonplace. The most basic differences between single crystal and polycrystalline materials are to the presence of grain boundaries and to a randomization of the orientation of individual crystals. Though microscopically their properties are anisotropic due to preferred slip directions, etc., this randomization makes them appear macroscopically to be isotropic. The influence of the grain boundaries on properties can also be significant but how this is so depends on the exact conditions of deformation and on the particular material. At low temperatures grain boundaries act as barriers to dislocation motion, thus strengthening the material. At elevated temperatures grain the opposite is true, grain boundaries can lower strength by providing an alternate path for both diffusion and dislocation motion. Also, impurities tend to segregate to grain boundaries, altering the properties of the grain boundary (and thus the material) in a number of ways depending on the nature and concentration of the impurity.

Two classic papers on the relationship between grain boundaries and strength were published independently by Hall [1] and Petch [2] around 1950. Hall and Petch studied different behaviors but arrived at essentially the same conclusion: that the grain size dependence of yield strength can be described by the equation

$$\sigma_y = \sigma_0 + \frac{K}{\sqrt{d}}$$

Hall-Petch
Relationship

where K is a constant and d is the mean grain size, that the grain size dependence is related to the length of a slip band, and that the maximum slip band length is determined by the grain size.

Hall: In 1951 Hall published three papers describing a study on the factors influencing the mechanical properties of mild steels. In the earlier papers Hall suggested a theory for the relationship between the upper yield point and grain size. Large grains tend to allow a plastically deformed nucleus to grow more rapidly which leads to the more rapid formation of Lüders bands. He expected that as grain size increased the differences between the upper and lower yield strength would diminish.

In the third paper Hall [1] explained the relationship between grain size and the lower yield point. It was well known that when dislocations encounter obstacles the dislocation's motion is impeded, causing the stress required to continue the deformation process to increase. Grain boundaries constitute such an obstacle. As dislocations pile up behind the grain boundary the stress concentration at the tip of the slip plane increases and will eventually cause the grain boundary to yield (as if grain boundaries constitutes a sort of shell, of film, surrounding the grain). When this happens deformation is transferred to the next grain. His experimental results showed that

$$\sigma_{LYP} = \sigma' + \frac{k_H}{\sqrt{d}}$$

Hall

where d is the mean grain size and σ' is the yield strength of a single crystal. Hall explained that the stress at some point ahead of the first dislocation in a pile up was analogous to Griffith's [3] analysis of the failure of brittle materials. (There is a critical stress above which a crack will grow and this stress is proportional to the inverse of the square root of the length of the crack.) The length of the slip band is analogous to the crack length and defines a critical stress required to punch through a grain boundary. The maximum length of a slip band is related to the mean grain size.

Petch: Petch’s study [2] focused on the brittle failure of steels. It was known that coarse-grained steels were more prone to brittle failure than were fine-grained materials. He had hoped to "throw a light on the whole subject of brittle failure" by measuring the variation in cleavage strength with ferritic grain size. Mild steel, ingot iron and spectrographic iron was tensile tested at liquid nitrogen temperatures to obtain brittle fracture. Special care was taken to prepare specimens free of surface (Griffith) flaws and that the testing machine provided true axial loading. Cleavage strength and fracture stress were measured. Petch's results for cleavage strength, σ_c, obeyed the following equation

$$\sigma_c = \sigma_0 + \frac{k_P}{\sqrt{l}}$$

Petch

where l is the mean intercept length (proportional to grain size) and σ_o and k_P are constants. Some plastic deformation was observed but was had a negligible effect. Petch discounted the possible role of Griffith flaws which can be associated with precipitates since Griffith theory would require a relationship between grain size and precipitate size instead of grain size. Besides, he doubted it would work since the same behavior was also observed in recrystallized zinc. However, the tip of a dislocation pie-up could be analyzed as a Griffith flaw. The tensile stresses at the end of a pile-up can be quite high

$$\sigma = \alpha n \tau \ldots$$

where ″is a constant, *n* is the number of dislocations in the pile-up and *J* is the shear strength. It was necessary to be able to relate *n* to grain size.

Petch argued that Frank-Reed loops could produce many dislocations on a single slip plane. These dislocations would pile up at opposite ends of the grain and the maximum value of *n* would be obtained when the Frank-Reed source is at the center of the grain. For a given stress, an array of *n* dislocations could pile up over half the grain diameter. An internal stress was incorporated in order to account for interactions with dislocations on other slip planes and the shear stresses were equated to normal stresses and the Petch equation above was obtained.

There was excellent agreement with Hall's results. Even the relationship between the upper and lower yield strength that Hall expected to see was confirmed. (The difference between them decreased to zero when grain size increased to 0.04 mm.) The similarity between the behaviors of cleavage and yield strength was attributed to the fact that yield strength involves shear stresses while cleavage involves normal stresses. Petch went on to note that carbon content did not effect the grain size dependence. However, the increased amount of pearlite did increase the rate of strain hardening.

Grain Boundaries: Hall knew that grain boundaries could act as barriers to dislocation motion, but he also viewed a grain boundary as a sort of "film". If the properties of the grain boundary were known then a more complete analysis of their strengthening effect could be made. Grain boundaries are simply planar defects between adjacent grains, each grain having different crystallographic orientations. Low angle grain boundaries occur when the miss-orientation of the lattices of adjacent grains is less than 15°. These boundaries can be modeled as an array of edge dislocations. The structure of high angle grain boundaries is more complex.

On of the other characteristics of grain boundaries is that they tend to attract impurities. In a sense, this might be thought of as a film which can either strengthen or weaken a material. For example, sulphur segregation to the grain boundaries in stainless steel causes it to become brittle and fail intergrannularly. Small amounts of bismuth in copper segregates to grain boundaries causing it to become brittle. Grain boundary segregation of impurities, however, does not always embrittle a material. For instance, pure Ni_3Al intermetallic is already brittle and prone to intergranular failure. Attempting to cold roll this material will produce a granular, sand-like material. However, manganese and hafnium are known to act as scavengers of sulphur impurities that segregates to the grain boundaries while boron and carbon promote grain boundary cohesiveness. Additions of around 0.1% boron increases the ductility of Ni_3Al to around 55%, ductility typical of many metals. Boron-doped Ni_3Al has even been demonstrated to be superplastic.

Grain Growth: Grain boundaries are lattice imperfections and as such increase the free energy of the material. There is a tendency to minimize the contribution from grain boundaries and this involves reducing the grain boundary area to grain volume ratio, something which happens as the grains grow larger. While other factors can cause grains to grow, this is essentially the process involved in normal grain growth (also called ideal grain growth). Non-ideal grain growth occurs when grain growth is inhibited by the presence of a second phase or is restricted by the edges of the specimen. Cold work, pressure, magnetic fields, etc. also cause grain growth to deviate from the ideal case.

While the rate of grain growth is determined by a number of factors it all boils down to the frequency at which atoms jump across the grain boundaries and to the abundance of these boundaries. The process of atoms crossing the boundary sounds a lot like diffusion, which involves thermally activated jumps of atoms into neighboring, unoccupied, lattice sites. Therefore, one would expect diffusion to be a significant factor in the growth rate. Also, the rate of grain growth is higher for fine-grained materials. Thus the initial growth rate would depend on the initial grain size. Finally, there are several geometric factors to consider, but for the simple cases the following expression is often adequate to represent grain growth

$$d^g - d_0^g = B\exp\left(\frac{-Q}{RT}\right)t$$

where d and d_o are the final and initial grain sizes, respectively, B is a constant, Q is the activation energy which is usually close to that for diffusion, and g is a constant. For normal grain growth g is equal to 2.

Preparation

1. What is a slip band? Deformation band? Slip line?

2. What is so "magic" about the value of the exponent in the Hall-Petch equation? (Should the Hall-Petch relationship hold for all materials?)

3. Would you expect the Hall-Petch relationship to hold for thin walled tube or thin sheet made from a coarse-grained steel?

4. At high temperatures, increasing the grain size causes the yield strength to increase. This is opposite of what the Hall-Petch relationship predicts. Explain.

5. At extremely small grain sizes (less than 100 nm) the yield strength decreases as the grain size decreases. Comment.

6. How would you go about measuring and reporting the average grain size of a moderately cold worked material?

7. What is the typical range of yield strength, UTS, and ductility of the material that we'll study in this experiment?

8. In general terms, what is yielding? Define and note the differences between yield strength, yield point, upper and lower yield point, offset yield strength and the limit of proportionality.

Safety Considerations

This experiment involves heat treating brass, tensile testing, hardness testing the specimens and mounting and polishing them for metallographic observation. Extreme care should be exercised during the heat treating phase of the experiment as the temperatures can be quite high (800°C) and therefore poses severe burn hazards to personnel and fire hazards to the building. Tensile testing poses additional hazards due to the high forces, to 50 kN, involved, the delicate nature of the extensometer, and to the sudden fracture of the specimens. Slight hazards are associated with polishing and grinding the specimens. Novices invariably end up having a specimen or two snag the polishing cloth, sending their specimen flying across the room. The etchant which is used, ferric chloride plus hydrochloric acid, is not particularly aggressive but should be respected. The mounting presses are fully automatic and if the proper procedure is followed should pose not hazard to the students or the equipment. Operating instructions for the presses are kept nearby.

Chemical Hazards

Moderate. Etchants containing hydrochloric acid and ferric chloride are used. Appropriate personal protective equipment should be worn and the etchants should only be used in a safe out-of-the-way part of the laboratory which is set up for only etching the samples. Proper technique should be used for etching the specimens, washing the specimens and disposing of the etchant. MSDS's for each of the etchants or the components of each etchant are available. MSDS's are also available for the polishing and grinding materials.

Physical Hazards

The potential for very serious burns exists. Temperatures approaching 800°C are used during these experiments. At these temperatures one can easily be burned while loading and unloading specimens from the furnaces, even if the hot specimens and furnace are not touched. It will be important to wear heat resistant gloves and to use long tongs. One should also take care to prepare a clear area to work, have an emergency procedure in place in case hot specimens are dropped on the floor, etc. It would be a good idea to rehearse the procedures for handling hot specimens.

Hardness testing poses very little hazard if proper testing procedures are followed. Using the proper anvil and indentor and a clean specimen will minimize the chance of damaging the equipment or injuring personnel.

Tensile testing machines can generate tens of thousands of pounds force. Be very careful when installing a specimen and stay back when the test is running. Brittle specimens and composites tend to send small debris flying about the room when the specimen breaks. Compression testing any material or structure poses the same hazard. If these types of specimens are used or if you plan to do a compression test then either a scatter shields should be installed on the load frame or everyone should be wearing safety glasses.

The polishing wheels can spin at several hundred rpm. Take care during the grinding and polishing phases of the experiment that your fingers or the specimen do not get trapped between the spinning wheel and the bowl. Also, take a moment to bevel both the tops and bottoms of the specimens to remove sharp edges that might snag the polishing cloth or grinding paper, get ripped out of your hand, cutting your fingers and sending the specimen flying across the room.

The mounting presses are fully automatic. Simply follow the established procedures and there should be no problem.

Bio-hazards

None.

Radiation Hazards

None.

Protective Equipment

Recommended: safety glasses are recommended during the polishing/grinding and hardness testing phases of the experiment. The use of protective coverings for the floor and counter tops is also recommended.

Required: safety glasses, heat resistant gloves and long tongs for the heat treatment phases of the experiment. Safety glasses and chemical resistant gloves must be used when etching the specimens.

Waste

Pour used etchant back into the original bottle. The dilute ferric chloride etchant which was washed from the specimen can be poured down the drain. Other disposal procedures may be required if etchants other than ferric chloride are used.

Used specimens can be recycled as scrap copper/brass.

Materials

The material tested in this experiment is the C26000 alloy, also known as cartridge brass. Its nominal composition is 70% copper and 30% zinc and it is a single phase ("-phase) alloy which tends to form twins. The material used in this experiment is in the half-hard condition.

Procedure

Given what you know about the grain growth behavior of this material, select annealing times and temperatures that will produce the desired grain sizes. For maximum control in this experiment, use the same temperature for all anneals.

Anneal all of the specimens and coupons in air. Make sure the tensile specimens are well supported so that they don't sag during the anneals. Also, make sure the coupons are heat treated in exactly the same manner as the tensile specimens. The method of quenching may not be important, but the same method should be used for each specimen and coupon.

Hardness test each specimen in the shoulder section and then tensile test it. Use the same testing procedure for each specimen. From the results determine the yield strength, tensile strength, ductility and other properties.

Prepare specimens for metallography using the coupons. The procedure one uses for brass is given in the appendix.

Examine the microstructure of each metallography specimen. First, make sure the specimen had recrystallized fully. Next, capture images to put in the report, shared with colleagues, or to measure grain size.

Measure the mean grain diameter for each metallography specimen using the mean lineal intercept method. These measurements can be made directly from the micrographs or straight from the specimen using a metallograph and a measuring reticle. This procedure is described in an appendix.

Analysis

Grain Growth Kinetics: Plot grain size -vs- annealing time. Determine the value of the grain growth index. Was ideal grain growth observed? Assuming a typical value for the activation energy determine the value of *B*. How do these results compare to those from a previous grain growth kinetics study?

Yield Strength: Plot yield strength -vs- grain size and in a manner that illustrates the Hall-Petch relationship. What is the value of *K*? Does the yield strength vary with the inverse square root of the grain size? Does this material obey the Hall-Petch relationship?

Other Properties: Other properties might also show a grain size dependence. Analyze the grain size dependence of UTS, ductility, strain hardening exponent, etc.

References

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2. N.J.Petch, J. Iron and Steel Institute, pp. 25-28, May 1953.

3. A.A.Griffith, Phil. Trans. Roy. Soc., Ser A, vol. 221, pp. 163-198, (1920).