RECOVERY, RECRYSTALLIZATION, AND GRAIN GROWTH

Introduction

Imagine basing a major industry on a few substances for which you had only the most superficial understanding. Now image sustaining this industry for over 5000 years. This essentially characterizes the metals industries from the time of the establishment of the earliest cultures up to the end of the Dark Ages. Up to the decline of the Roman empire there were only seven metals: gold, copper, silver, lead, tin, iron and mercury available to metal smiths. But this limited selection of metals was not the reason for such slow progress in the field. Instead, people simply did not understand the processes they employed to refine and work their metals and these processes were virtually uncontrollable due to the lack of even the most basic knowledge of chemistry or even the ability to control the temperatures of their furnaces. Ancient metallurgical practices were based on trial-error, tradition, superstition and myth. And while the quality of certain metals could be high it could only be produced in small quantities. For instance, a good day's work in a major Roman smelting facility would yield only 50 pounds of iron. Still, the ancients possessed a high level of

Figure 1. Part of a four-part necklace of gold leaves and precious stones, from the Royal Graves at Ur; ca. 2600 BCE. [1] This is an excellent example of the quality of metal working done almost 5000 years ago. The gold, found in its native metallic form, was already of relatively high purity and required no further refining. Metal working was limited to simply hammering these nuggets into thin sheets. Pure gold is very ductile.

technical skill and were able to work their seven metals to produce art, farm tools, weapons and many other useful and decorative items including the famous and still intriguing, metallurgically speaking, swords of Damascus, the 6 ton, 23 feet tall, 99.7% pure iron pillar of Delhi (built in 310 CE and is still standing) and King Solomon's 200 ton bronze statue.

Progress was slow and intermittent. To start with, the use of metals usually lagged the development of farming practices in a culture by 2000 years. And while gold and native copper had been used as early as 5000 BCE it wasn't until around 2000 BCE that they began to process ore and it took another half a millennium before they began to intentionally smelt two metals, copper and tin, to make bronze. The quenching, tempering and carburizing of iron had been employed, though not widely, as early as 900 BCE but as late as the dark ages, by which time iron had virtually replaced bronze in weapons and armor, swords and edged weapons were still very unreliable. In those days proven swords were priceless and a simple sword was expensive, costing the equivalent of three cows [1]. Legends and historians both speak of famous swords, but in their time the effectiveness of these weapons was attributed more to the magic runes on the blade and to blessings from the gods than to skillful metal working and perhaps luck. It is no wonder that metal working practices had changed so little from those practiced over a thousand years earlier.

Civilization gradually developed the intellectual and technological tools to be able to ask, and answer, more interesting questions concerning the physical world. This produced an ever better understanding of the physical world, of physical processes, of the materials we use and, most people will agree, quality of life. Among the benefits of the rapid advances in mathematics, physics, chemistry that took place during the Renaissance were a larger selection of high quality metals. But it has been only in the last 150 years or so that we could finally see that, like our buildings and machines, the materials they were made of had their own well defined internal structure. In the 1860's Dr. Sorby, the inventor of metallography, showed us the microstructures of a number of metals while Bragg and his contemporaries at the turn of the twentieth century showed us that the structure of many solids on the atomic scale was crystalline. Finally, after thousands of years we could see how alloying and processing influenced the structure of metals and we could correlate properties with the structure, thus completing the structure-processing-properties triangle that is the basis of modern materials science.

Modern brasses (copper-zinc alloys) and bronzes (copper-tin alloys) are distant relatives of the metals whose usage defines the bronze age. Ancient bronzes were usually made by smelting native copper (naturally occurring metallic copper, typically greater than 98% pure) or copper extracted from the few available ores, with tinstone (pure tin was not available until late Roman times) and charcoal. They quickly found out that bronzes were much more difficult to hammer than the softer, more ductile copper. However, it could be worked while hot or after reheating. It could even be welded by hammering while the metal was hot. While these processes worked, one doubts if the ancients had any theories for why they worked and if they did they probably invoked the four elements and four qualities which were postulated by the Greek philosophers.

From many detailed investigations of the structure of metals and alloys we know that mechanical deformation hardens a metal by increasing its dislocation density. While dislocations are an essential part of the deformation process, indeed their motion accounts for most of the deformation, high dislocation densities make it more difficult for them to move freely. As a result, increasingly higher stresses are required to continue the deformation process. But as the dislocation density and stress continue to increase it becomes more likely that a crack will form and the part will fracture.

Heating this hardened material will decrease the dislocation density and can even cause a completely new, virtually dislocation-free, microstructure to form. The result is a softer material which one can continue working. Alternatively, one can hot work the material. During hot working the material does not harden as readily because the dislocation density is never very high, making much more deformation possible and reducing the chance that a crack will form.

Much like the metal working practices of the earliest metallurgists modern metal working processes often involve cycles of mechanical working and heating, only now with many refinements and with much more control over each step in the process. For instance, in modern mills it is common practice to reduce the amount of working with each cycle in order to obtain a more uniform grain size. This is because working the metal tends to deform the outer sections more than the in the center. Also, the more the material is worked the more readily it will recrystallize. The heat treating part of the cycle involves mainly time at an elevated temperature and in a controlled atmosphere but cooling and heating rates may also be significant factors. For example, rapid heating can cause leaded brasses such as the C36000 alloy to fracture due to the presence of significant amounts of undissolved lead which has a different coefficient of thermal expansion than the Cu/Zn phases. On the other hand, rapid cooling can preserve the microstructure of a material which existed at the furnace temperature while slow cooling can lead to extensive precipitation and coarsening of the microstructure. In many ways the old and new metal working processes are similar but now the processes are much more reliable, the results are much more consistent and of higher quality, and it is now possible to process tons of material at a time. Moreover, our knowledge of structure-property relationships allows us to readily adapt our metal working processes to meet new forming and properties requirements or to deal with new alloys.

This experiment deals with three very specific types of heat treatments. Each is concerned with producing the desired properties by means of producing the required microstructure. These heat treatments, their objectives and the resulting microstructure are:

Stress Relieving	Stress relieving reduces or eliminates residual stress, thereby reducing the likelihood of failure during service, usually by stress-corrosion cracking. It can also be used to improve dimensional stability. Stress relieving temperatures are below the recrystallization temperature. While stress relieving near the recrystallization temperature will require shorter times and will be a more economical process there will be some loss of strength. Using lower temperatures and longer times will preserve strength and will even increase it slightly if the material had been severely cold worked.
Recrystallization	Recrystallization is characterized by a rapid change in tensile properties and the formation of a new, strain-free microstructure. Between 35 and 60% cold work is required before recrystallization can occur with increasing amounts of cold working lowering the recrystallization temperature.
Softening, annealing	Softening is achieved by heating to well above the recrystallization temperature and holding for whatever time is required to complete the recrystallization process and obtain the desired grain size. Annealing is often used when the material will receive further cold working.

The main difference between ancient and modern metallurgy is not the number of different metals we refine, the quality or quantity produced or the high degree of control over the processes we employ. It is the fact that we know so very much about the structure of our materials and we appreciate its relationship to properties. We tend to explain properties in terms of structure (this definition of structure includes chemistry) and we design our processes to produce the desired structures.[1]

Objective

The two objectives of this experiment combine the practical: obtaining information of immediate interest to a process engineer, and what many people would call the academic: improving our understanding of the more fundamental nature of engineering materials. In this experiment tensile tests will be conducted on a series of specimens which had been annealed at different temperatures for one hour. The basic tensile properties are then measured and evaluated in order to determine the temperature ranges for stress relieving, recrystallization and annealing. Further support for the determination of these temperature ranges is provided by examining the microstructures. This would satisfy the first objective. The second objective will be satisfied by correlating these heat treating temperature ranges with specific microstructural processes, thereby establishing the interrelationships between a material's structure, its processing history and its properties.

Preparation

Before actually starting the experiment you should pause to consider each aspect of the experimental procedure and to try to anticipate the results. This will help ensure that the experiment goes well. The following questions should get you started.

1. Look up the mechanical properties of the brass in your reference books. Note that you will need to consider the full range of strength, ductility, hardness, etc. Compare the magnitudes of the range of yield strength, UTS and ductility. Which one is the greatest?

2. The preparation section of the "Basic Mechanical Properties" experiment poses a number of questions to help one plan and execute the hardness and tensile tests. Review these questions.

3. Find a Cu-Zn equilibrium phase diagram and draw a vertical line at the composition of the alloy you will be testing. Note the melting point, solubility limits and the types and quantities of phases present at the temperatures used in this experiment. Make a rough estimate of the recrystallization temperature for this alloy.

4. Locate micrographs of cold worked and annealed brass and learn to recognize grains, twins, deformation bands. Make a sketch of microstructures which contain these features.

---

[1] The ancients may have also placed some value on structure-related qualities such as color and uniformity. The significance of this uniformity can be seen in the patterned steels of the Norsemen and early Europeans and west Asians. The patterns obviously meant something to them but it seems to have been largely a concern for aesthetic and magical properties. Some medieval smiths believed that after forging the steel it had to be quenched in the urine of a red-headed boy or that of a three-year old goat fed only ferns for three days. Even in the hardening of Damascus steel (bulat), a legendary and very finely patterned steel, the following account of the hardening process reveals the mystical qualities of their metallurgy: "The bulat must be heated until it does not shine, just like the sun rising in the desert, after which it must be cooled down to the color of king's purple, then dropped into the body of a muscular slave... the strength of the slave was transferred to the blade and is the one that gives the metal its strength" [2].

5. What exactly is a grain, a twin and a deformation band?

6. What is the role of diffusion in the heat treatments applied to the alloy studied in this experiment?

7. What is the role of cold working in the recovery and recrystallization processes? Is it important in the grain growth process?

8. Why do grains grow? What is the upper limit for grain size? Lower limit?

Materials

The alloy investigated in this experiment will be either 70/30, 60/40 (Cu/Zn) or free-cutting brass. The 70/30 alloy is formally known as the C26000 alloy and informally by its common names: " brass, 70/30 and cartridge brass. It has a single phase microstructure (", fcc) from approximately room temperature to its solidus temperature. The UNS designation of the 60/40 brass is C28000. Its common name is Muntz metal but is occasionally called "/$-brass. It is a two-phase alloy consisting of varying proportions of the " (fcc, copper-rich) and $ (bcc, zinc-rich) phases from below room temperature up to approximately 900°C. The composition of the free-cutting brass is 61.5Cu-35.5Zn-3Pb. (Its UNS designation is C36000.) The lead is added to improve machinability but it severely limits this alloy's ability to be cold worked. Its microstructure consists of three phases: ", $ and undissolved lead and its solidus temperature is 885°C.

Whichever alloy is used, the material will already be machined into tensile specimens and heat treated at various temperatures for 1 hour. Note the exactly which alloy you are using, its original (as received) condition, and the details of the heat treatments. The specimens used for metallographic examination will already be mounted, polished, etched and ready for viewing in the metallographic. The details of the preparation of the metallography specimens are not important in this experiment.

Equipment

The following is a generic list of the types of equipment that may be used to perform this experiment. Make sure you have everything you need before starting and note exactly what types of equipment (manufacturer, model, specifications, etc.) you are using.

1. Rockwell-type hardness tester
2. Tensile testing system
3. Computer and data acquisition system to record the force and elongation data. If a computer is not available then a strip chart or flatbed recorder can be used.
4. Calipers to measure the specimens
5. Metallographs

Safety

During this experiment high forces are generated by the tensile testing machine. It has pinch zones large enough to trap a finger, a hand and even an arm. Injuries, if they should occur, could be quite serious. A detailed operating procedure is included in this laboratory manual. Read it before you start using this equipment and refer to it during every step of the experiment. Be especially careful when installing a specimen, stay clear while the test is running and keep in mind that some types of specimens may shatter when broken.

Chemical Hazards	Normally none, but this will depend on the materials the specimens are made from. Specimens are normally made of steel, copper, brass, aluminum alloys or other conventional structural materials.
Physical Hazards	The tensile testing machine 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 type of specimen 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.
Biohazards	None.
Radiation Hazards	None.
Protective Equipment	Recommended: safety glasses Required: safety glasses and/or scatter shields if compression testing is done or if brittle materials or composites are tensile tested.

Procedure

Examine the experimental equipment and find out what each instrument and tool does and how each works. Make sure everything is working properly and if possible try a couple of dry runs of the experiment.

Hardness test each tensile specimen. Do not perform these tests on the gage section of the specimens. You don't want to damage the section which you will be tensile testing. Test the heads of the specimen instead. Also, make sure you are using the appropriate hardness scale.

You may have to convert several of the hardness readings to another hardness scale so that you can plot the data. Remember that these conversions are approximate and that when you report the converted values you also have to report the original values. Refer to the appendix on hardness testing to find out how to correctly report hardness numbers.

Tensile test each specimen to failure using the protocol provided by your instructor. Preview this protocol before you use it and note details such as the crosshead speed and the data acquisition rate. If the system is capable of providing a report along with the data then make sure you get a copy of this report.

Polished and etched specimens of the materials you just tested will be provided by your instructor. Your instructor might also provide micrographs. Examine the microstructures using the metallographs and the micrographs. Make notes of the prominent and distinguishing features in each specimen. Try to identify each specimen in terms of the type of heat treatment it has received:

stress relief, recrystallization, anneal.

Results

Construct an engineering stress-strain curve showing the data for all specimens tested. Using these graphs or a spreadsheet containing the data measure the following properties:

1. Young's modulus
2. Yield strength
3. UTS
4. Ductility in terms of elongation to failure. If round specimens were used then you should be able to measure final reduction in area.
5. Modulus of toughness.

All of the above properties should be organized and presented in a table and each property should be plotted to show how each is effected by the heat treatments. Summarize the results, validate the results, point out significant features, determine temperatures.

The micrographs are data, too, and must be presented to the reader who might not have as much experience with this alloy as you have. Point out those features which are relevant in this experiment and describe the trends you find. You might try presenting this data as if they represent observations that were functions of time, as if higher temperatures correspond to longer heat treating times. But be careful how you do this so that you don't give the reader the impression that you have confused the two.

Discussion

You have plenty of data to help you meet your objectives. You have already described the trends in tensile properties and should have noticed that transitions in each property occur over the same temperature range. It should be easy to determine the stress-relieving, recrystallization and annealing temperatures.

If you used the same scaling on the temperature axis and if your graphs are all the same size then you will clearly see how one property relates to another. For instance, an increase in hardness is accompanied by an increase in strength but by a decrease in ductility. This illustrates one of the most common compromises an engineer must make when specifying a material for an application.

It might be interesting to consider how much each property is effected by the heat treatments. Compare the ranges of each property in relative terms to see which is the most effected. What would this mean to an engineer who might need to purchase ten tons of this alloy?

Your observations of the microstructures provide the critical link between processing and properties. You have described the trends in the mechanical properties and have determined the temperature ranges for the different types of heat treatments and now with the microstructures you can explain what is happening. In doing so you might try recasting the stress relieving, recrystallization and annealing heat treatments in terms of recovery, recrystallization and grain growth. Looking for links to a more fundamental process you might compare these temperatures to the melting temperature, or solidus, of the alloy.

Conclusion

You have had an opportunity to investigate both the mechanical properties and the microstructures of a series of heat treated specimens which were all made from the same alloy. From these two perspectives on the effects of the heat treatments you should be able to determine the temperature ranges for stress relieving, recrystallization, annealing treatments. While this satisfies the first objective of this experiment it is really only a superficial analysis of what is happening. If you step back and take a broader view of these results you should clearly see that there is a definite relationship between properties, processing and structure. Your conclusions on this matter will satisfy the second objective.

References and Other Resources

1. Leslie Aitchison, A History of Metals, Interscience Publishers, Inc., New York, (1960).

2. O.D.Sherby and J.Wadsworth, Scientific American, vol 252, n.2, pp112-120, (1985).

3. J.F.Shackelford, Introduction to Materials Science for Engineers, 4th edition, Prentice-Hall, Inc., New Jersey, pp. 240-242, (1996).

4. W.D.Callister, Materials Science and Engineering, An Introduction, John Wiley and Sons, Inc., New York, 3rd edition, pp.165-175, (1994).

5. D.R.Askeland, The Science and Engineering of Materials, PWS Publishing Co., Boston, pp.177-196, (1994).

6. ASM Metals Handbook, Mechanical Testing, 9th edition, ASM International, Metals Park, OH, vol.8, pp. 17-51 and pp. 69-113, (1986).

7. ASM Metals Handbook, Properties and Selection: Nonferrous Alloys and Pure Metals, 9th edition, ASM International, Metals Park, OH, vol.2, pp. 275-382, (1986).

8. ASM Metals Handbook, Metallography and Microstructures, 9th edition, ASM International, Metals Park, OH, vol.9, pp. 399-414, (1986).

9. Annual Book of ASTM Standards, Section 3, American Society of Testing and Materials, Philadelphia, PA, vol. 03.01, (published annually).

10. Materials Science and Engineering Handbook, 2nd edition, J.F.Shackelford, ed., CRC Press, Boca Raton FL, (1994).

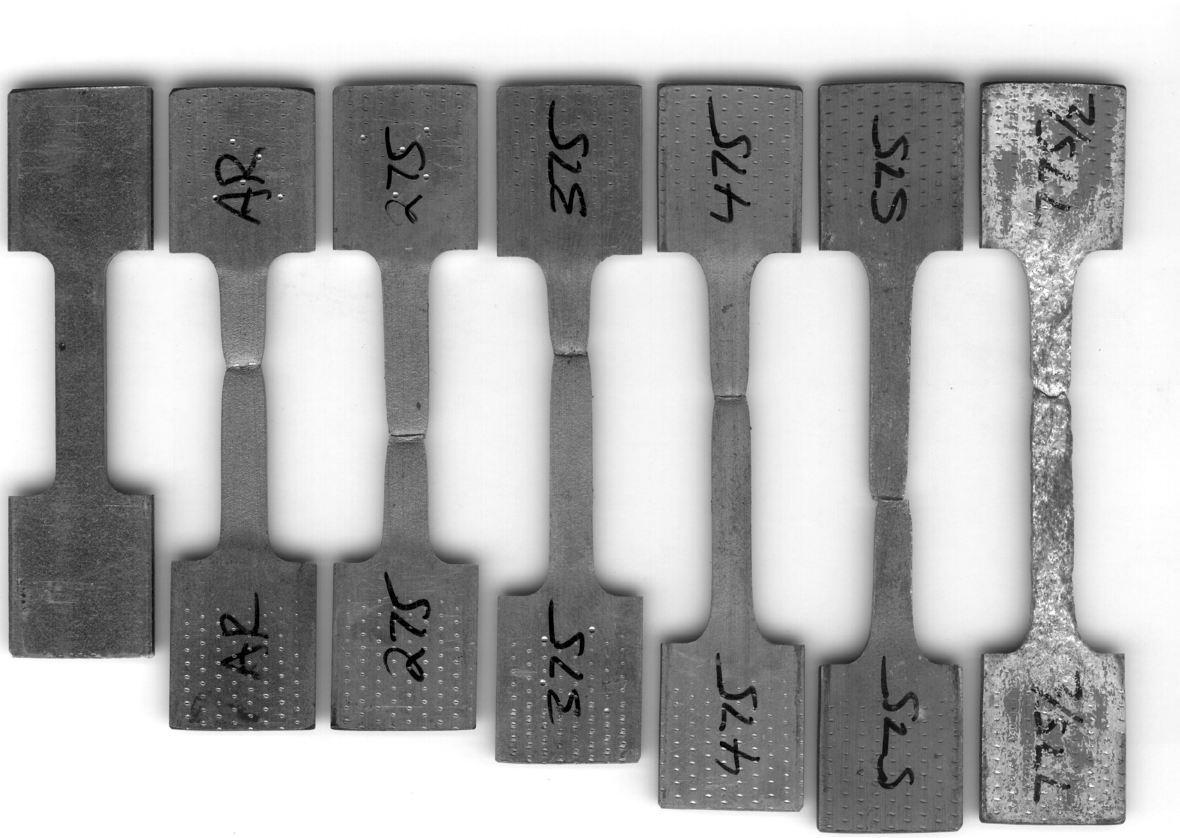

Figure 2. Tensile tested specimens made of 70/30 brass. The heat treatment on each specimen, from left to right was: untested, as received (full-hard), 275°C, 375°C, 475°C, 575°C for 1 hour and 775°C for 2 hours. Note the roughness of the gage section of the right most specimen. This and its lower ductility than the 575°C specimen is due to the very large grain size, approximately 1 mm.