Washington University in St. Louis

MEMS 205 Mechanics and Materials Science Laboratory

Lab 2: Materials Processing

Section: Group D (Tuesday 12 PM)

Lab Instructor: Dr. Sellers

Experiment Date: Tuesday 2/4/2020

Report Submission Date: Tuesday, 2/11/2020

We hereby certify that the lab report herein is our original academic work, completed in accordance with the McKelvey School of Engineering and Undergraduate Student academic integrity policies, and submitted to fulfill the requirements of this assignment.

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Abstract

We seek to document the effects of material processing on the hardness of copper and of steel from the stock of these metals recently acquired by NASA. We examined the percent cold work (%CW) to the hardness of copper, 1018 steel and 1045 steel by using a Rockwell hardness machine and furnace and a cold rolling machine. The Rockwell hardness machine consisted of an anvil for the specimen and indenter to measure the hardness. The furnace was used at a temperature of 400˚C, 550˚C, and 910˚C to heat up the specimen. The cold rolling machine consisted of two rollers for the specimen to slide through. At ambient temperature we saw that copper had a hardness of 74.7 HRF, 1018 steel had a hardness of 82.2 HRB and 1045 steel had a hardness of 90.8 HRB. After we annealed copper at 400˚C the hardness of copper decreased. When we annealed both steel specimens at 910˚C, the hardness of the steel specimen increased. As the copper was cold-rolled we measured the hardness to increase with a decreasing slope as the percent cold work increased. We also saw that as copper was tempered at 440˚C it decreased the hardness rapidly but evens out over time. For both steel specimens, we saw that after 30 minutes of annealing at 550˚C it decreased the hardness of the specimen.

Introduction

Hardness is "a measure of a material's resistance to localized plastic deformation" [1]. The harder the material, the harder its surface is to scratch, similarly the hardness of the material is directly proportional to its tensile strength [1]. Therefore, the effects of materials processing on copper and steel will directly affect the applications for these metals in NASA's approaching BioSentinel project. There is no universal technique to test hardness, but the Brinell and Rockwell hardness tests are commonly applied. This experiment uses the Rockwell hardness test to determine the effect of different forms of material processing on a material's hardness.

The Rockwell hardness test is a two-stage process, normally automated by a Rockwell testing machine (Fig. 1). A tungsten carbide ball or conical diamond indenter, depending on the specimen that is being tested, is pressed into the subject by a 10 kg load, called the minor load, then, the load is increased to a higher load, called the major load. The difference in penetration caused by these two loads is quantified and outputted as a hardness value, which depends on the appropriate scale for a specific specimen. Each separate scale ranges in Rockwell hardness values from 0, softest, to 130, hardest, but is only accurate between 20 and 100. The separate scales range alphabetically from A to K, with A corresponding to the lowest hardness values and K with the largest. These scales also utilize a unique indenter load combination and specific indenter depending on the expected hardness of a material. This lab utilizes scale B and scale F, for steel and copper specimens respectively. Scale B is based off the 1/16-in. tungsten carbide ball indenter with a 100 kg load, whereas scale F is a 60 kg load with the same indenter. The correct alphabetical scale, corresponding indenter, and applied loading must match the test specimen so the resulting measurement produces hardness values between 20 and 100.

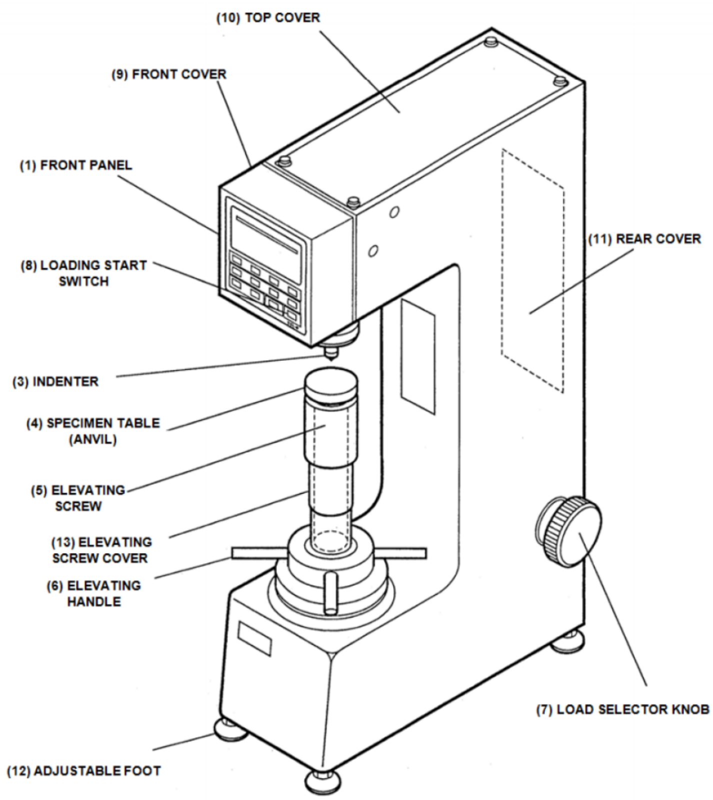


Fig 1. Rockwell Hardness testing apparatus without an included specimen on the table [2].

There are various material processes that can affect materials hardness. Cold rolling, annealing, heat treatment followed by quenching, and tempering are material processing techniques that this experiment utilized to affect the hardness of a metal. Cold rolling is when a metal is plastically deformed by compression as it passes between a hard-cylindrical roller and a hard-bottom plate (Fig. 2). The extent to which metal has been cold worked during cold rolling is expressed as "percent cold work" ($\%CW$) and is calculated as:

$$\%CW = \frac{A_0 - A_d}{A_0} \times 100. \tag{1}$$

A_0 is the cross-sectional area of the metal before cold working and A_d is the cross-sectional area after the cold working. Cold rolling causes a decrease in the cross-sectional area of the metal, increasing $\%CW$ [5]. The effect of the cold rolling process is known as strain hardening [4]. Through this process, a metal becomes tougher as dislocations are created and collide with one another, which is the mechanism of plastic deformation. This interrupts dislocation propagation, thereby inhibiting plastic deformation and increasing the hardness of the metal [5]. However, once the material reaches a critical $\%CW$, the hardness of the will no longer increase as much with increasing %CW [5]. In addition, from the Hall–Petch relationship we know that reducing the grain size to near the amorphous limit of a material will actually cause grain boundaries to slip as opposed to preventing dislocation motion we observe a weakening in the material [6].

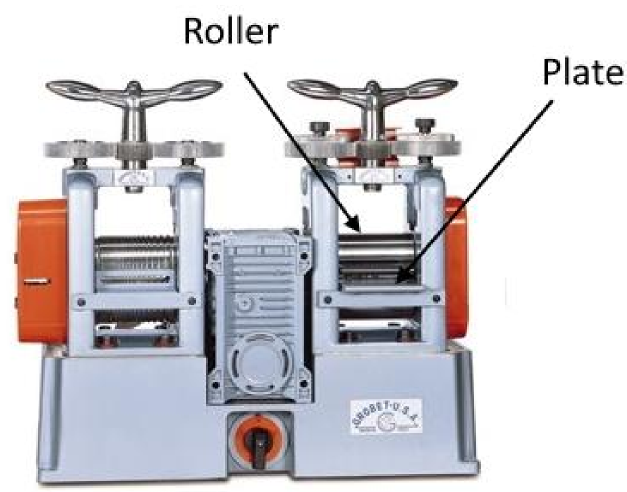


Fig 2. Cold rolling machine diagram with the cold rolling mechanism on the right side [3].

As dislocations pile up on one another during cold rolling, strain energy is stored within the metal's lattice. As more internal energy is stored, tensile strength increases but ductility decreases [1,5]. This reduces the overall toughness of the material and amount of plastic deformation that can occur before catastrophic failure results.

The metal can be heated up to increase ductility and decrease hardness. This allows dislocations to begin to move, allowing some dislocations to annihilate one another and others to reorient, which lowers the overall internal strain energy. At this point, diffusion encourages nucleation within individual crystals, annihilating dislocations and producing a lattice of similarly sized crystals with low dislocation densities. Finally, nucleation becomes the dominant force, causing larger grains to absorb smaller grains. This reduces the grain boundary area, resulting in further reduction of the internal energy of the material [1].

Heat treatment of alloyed metals, followed by quenching, also produces hardness and high internal stress levels. The 1018 and 1045 steel stock, acquired by NASA, is a low iron-carbon alloy with weight percentages of carbon being between 0.022 and 2.14 wt%. As this supplied steel undergoes heat treatment, its microstructure experiences two-phase transformations before becoming liquid [1]. At room temperature, the microstructure of steel is ferrite with cementite precipitates. Ferrite is BCC iron with carbon interstitials as well as Cementite, which the molecule Fe_3C. As heat treatment temperature increases, the microstructure becomes Austinite, which is FCC iron with carbon interstitials. Just before melting a BCC structure is adopted, which is now termed delta-ferrite [7].

Heating steel to the austenite phase and then quenching it rapidly produces martensite. This transformation is seen in a time-temperature-transformation diagram (Fig. 3), which plots the microstructure of steed as it cools over time. Martensite is a body-centered tetragonal iron with carbon interstitials and is very hard. Quenching austenite does not allow time for diffusion to occur, so carbon is trapped in interstitial positions at low temperatures that normally cause cementite to form. These carbon interstitials make it difficult for dislocations to propagate, causing martensite to be very hard [7].

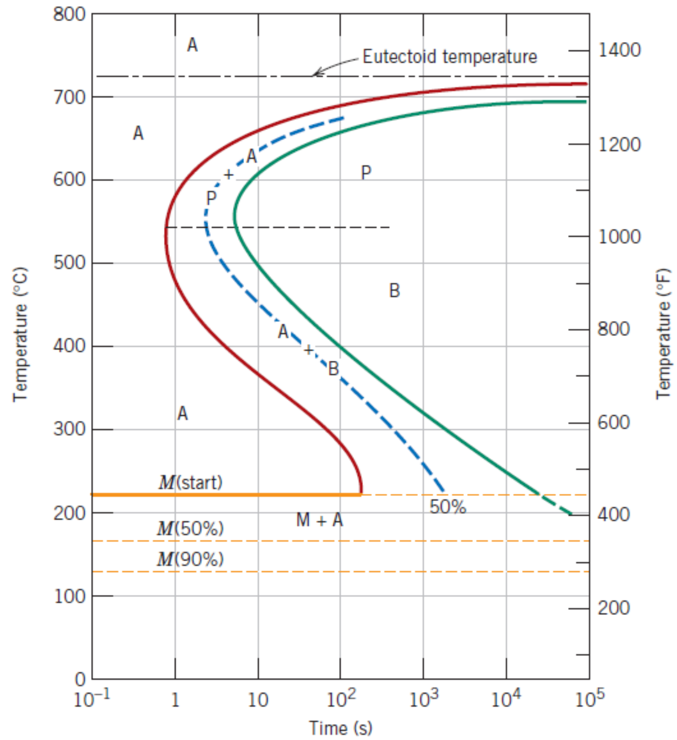


Fig 3. Time-Temperature-Transformation diagram of mild steel.[7].

However, martensite is often too brittle to be useful. Just as cold-worked metal can be softened with the annealing process, martensite is made less brittle with tempering. In tempering, martensitic steel is reheated to a temperature high enough so that diffusion becomes significant but low enough that austenite particles do not form. Diffusion allows cementite particles to form as body-centered tetragonal iron returns to the BCC. As a result, tempered martensite is much harder than ferrite, but much more ductile than non-tempered martensite, making tempered martensite a versatile material for NASA's use [7].

Cold worked copper can also be tempered to increase its ductility. Heat is applied, causing internal strain energy to decrease as the dislocation density decreases. Determining the relationship between tempering and hardness of copper will determine how NASA processes copper for use on flashing in the BioSentinel project.

Experimental Methods

To measure the hardness of the 110 copper bar, 1018 steel, and 1045 steel, a Rockwell hardness tester was used. The copper specimen was 1.085 in. by 3.295 in. by 0.125 in. and both of the steel samples were 1.705 in. by 1.705 in. by 0.120 in. While using the Rockwell hardness tester, it was important to make sure that the anvil was flat and the indenter was fully locked before testing. The 1/16 in. as ball indenter was used. For copper, the Rockwell hardness tester was set to 60 kgf, 1/16 in. on the front panel and the load selector knob on the right side of the machine. The steel was set to 100 kgf, 1/16 in., and 100 on the knob. Each piece was first tested under ambient temperature. Afterward, the 110 copper bar was put into an oven furnace at 400 ˚C for at least 15 minutes. Both pieces of steel were put into another oven furnace at 910 ˚C for at least 30 minutes. In both cases, tongs and gloves were used in order to not burn yourself. After the time was up the steels were quenched for at least 1 second and tested again for hardness. The copper was also removed, but allowed to cool naturally until it reached room temperature. We wiped off the water on each sample and sanded off the scale on the steel so that results were not affected by the presence of these substances. The steel was then put back in the oven furnace at 550 ˚C for another 30 min. While the Steel was in the furnace the copper was cold-rolled to reduce thickness. During this process, we made sure to push the specimen through the cold roller with a ruler in order to keep our hands a safe distance away from the machine. After each time through the cold roller, the handle was turned approximately a quarter of the way around to decrease the thickness. We made sure to have at least two data points between 0-10% reduction in thickness and then continued at periodic intervals to test the hardness until the sample reached 74.4 %CW. It was then cut into four equal squares using tin snips and put into a furnace for 1-20 minutes at 400˚C. Each specimen was taken out at different times between 0-20 minutes to test the hardness. While waiting for the copper to anneal, the steel was taken out of the furnace and quenched in water for at least 1 second again. The specimen was then sanded to remove any surface scale, then each specimen was tested for their hardness.

Table 1 Equipment used to measure impact energy

Equipment	Serial Number	Quantity	Description	Tolerances
Rockwell Hardness Tester	230690	1	LECO Code NO. 810-251A Model: RT-240	+/- 0.1
Electric Furnace	01509106001130503 0150913201130506 01529080011105909	3	Thermolyne, Thermo Scientific Model #: FB1315M	None
Cold Rolling Machine	707110008	1	Model: GL 100 Id: 62 L 017	None
Dial caliper	None	1	None	+/-0.001

Results and Discussion

There is a give and take in materials processing. To gain hardness or strength, one has to give ductility. Finding the right balance of hardness and ductility is critical to obtaining the best material for the task at hand.

Table 2 below includes the initially measured copper hardness, the hardness after the copper was annealed for 15 minutes at 400 °C, and the hardness of the specimen as %CW increased. Each hardness value is the average of three significant measurements under the same conditions. Early in the process, the team noticed that when switching the Rockwell Hardness Tester between the different scales for copper and steel, the first test value was significantly lower than the expected values and lower than subsequent measurements of the same specimen.

Table 2 Rockwell hardness of copper under specified heat treatment conditions. Under each condition, the hardness was tested in at least three different locations and the average value was calculated.

Copper Condition	Rockwell Hardness Test 1 (HRF)	Rockwell Hardness Test 2 (HRF)	Rockwell Hardness Test 3 (HRF)	Rockwell Hardness Test 4 (HRF)	Average Rockwell Hardness (HRF)
Initial Hardness	37.1	71.5	75.2	77.4	74.7
Annealing at 400 °C	11.5	21.9	20.1	25.1	23.4

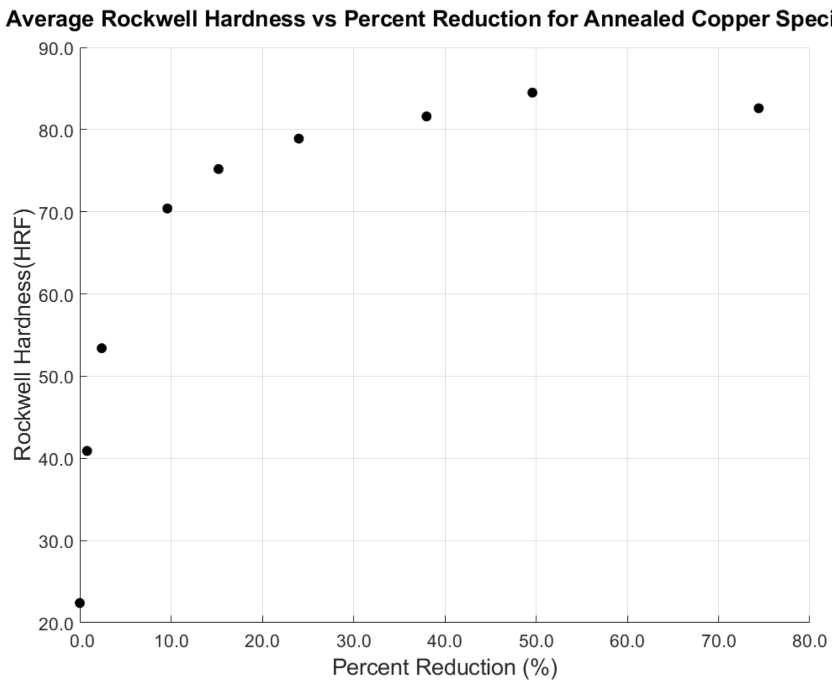
Table 3 represents the percent thickness reduction, or %CW, of the copper specimen with corresponding Rockwell Hardness measurements. The average of three measured values for Rockwell Hardness was taken for each %CW, excluding the first data point where only two measurements were taken. This data point only had two measurements due to a data processing mistake, with this data point not appearing in the final raw data sheet (App. A).

Table 3 Effect of cold rolling copper on the hardness of the copper. Average hardness was determined for each %CW from three unique hardness measurements.

Percent Thickness Reduction (%)	Rockwell Hardness Test 1 (HRF)	Rockwell Hardness Test 2 (HRF)	Rockwell Hardness Test 3 (HRF)	Average Rockwell Hardness (HRF)
0.8	36.9	44.9	-	40.9
2.4	53.9	52.9	53.3	53.4
9.6	70.0	70.3	71.0	70.4
15.2	75.4	74.0	76.2	75.2
24.0	78.7	78.5	79.4	78.9
38.0	82.2	82.6	79.9	81.6
49.6	85.3	84.2	84.1	84.5
74.4	83.0	82.5	82.4	82.6

Fig. 4 below represents the percent cold work, as defined by Eq. 1, which has a direct relationship to the hardness of the material. The more the copper was strain hardened the harder the material got. However, as can be noted the relationship was not linear. For the first few %CW, the copper hardness increased dramatically. Around 15-20% CW the hardness was not increasing as quickly. As we approach 40-50% CW, Fig. 4 shows the relationship beginning to flatten out. This plateau, in figure 4, leads to the idea that there is a point at which the %CW no longer has a significant effect on the hardness.

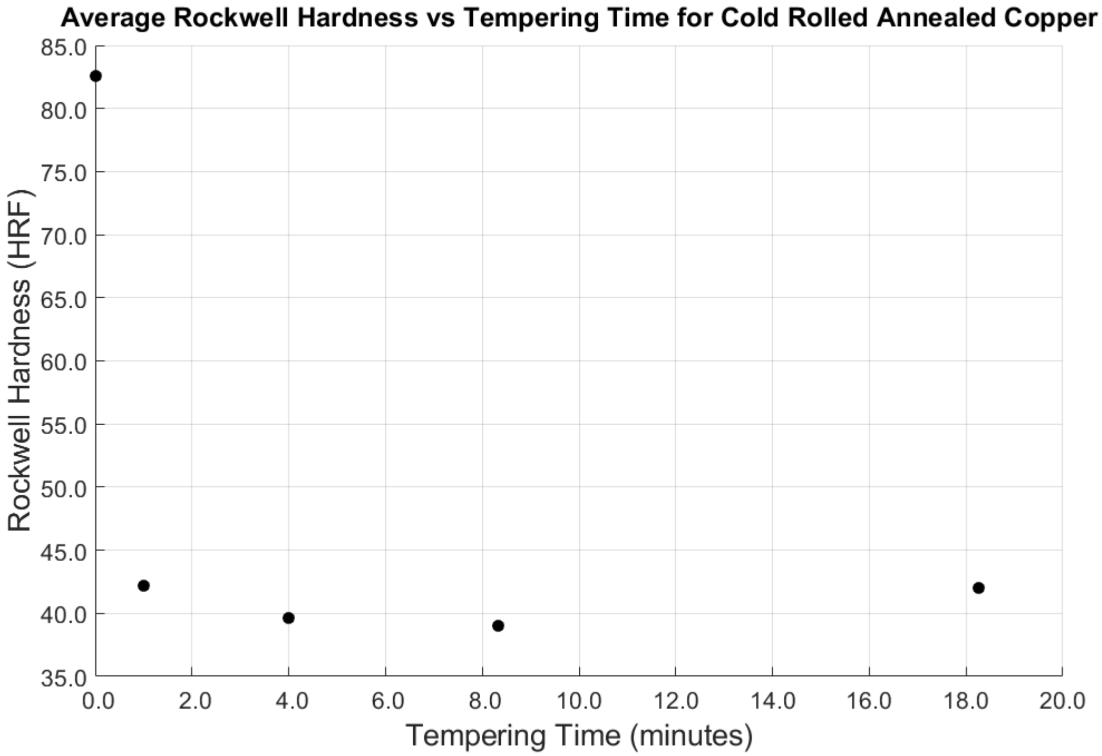
Fig. 4 Shows the relationship between hardness of the copper and %CW. The hardness of the copper increases dramatically with only a few % CW of the copper until around 40 %CW then the graph reaches a plateau.



The team observed that with 74% CW the hardness of the copper actually decreased. This is due to the fact that the more copper is cold rolled the harder it is to keep the specimen flat for testing hardness testing. For the 74%CW the team noted that the material was not flat for the the hardness testing. This can lead to a reading of hardness that is not correct. However, we also expect from the Hall–Petch relationship that after a certain point of reducing the grain size, copper will actually get weaker [6].

Figure 5 depicts the relationship that the tempering of the copper specimens has on the measured hardness. As expected, the hardness decreases with the amount of time the copper was tempered. This is because the internal strain from dislocation is decreased as dislocations annihilate. Similarly to cold rolling, we see that much of the change happens very quickly. With only one minute of tempering, the coppers' hardness has decreased by 48.9%. In one minute of tempering the hardness of copper has nearly halved. Thus, we see that any kind of material processing on copper has significant and immediate effects on the hardness of copper.

Fig 5. Shows the relationship of hardness to tempering time at 400 °C. The hardness dramatically decreases with only a small amount of tempering.



We also analyzed the effects that specific heat treatment material processing had on two different steel specimens. In Table 4 and Table 5 below the effect that the two different heat treatments have on the hardness of the steel can be seen. As expected, annealing and quenching the steel increased the hardness. While tempering the steel decreased the hardness.

Table 4 Effect of different heat treatments on 1018 steel. The initial hardness was measured and then the steel was annealed and tempered. As expected, annealing and quenching the steel increased the hardness. While tempering the steel decreased the hardness.

1018 Steel Condition	Hardness Test 1 (HRB)	Hardness Test 2 (HRB)	Hardness Test 3 (HRB)	Hardness Test 4 (HRB)	Average Rockwell Hardness (HRB)
Initial Hardness	52	80.7	82.4	83.6	82.2
Austenitic State at 910°C	84.8	90.2	87.7	N/A	87.6
Tempering at 550 °C	82.9	85.2	85.6	86.5	85.8

Table 5 **Effect of annealing and quenching on hardness along with the effect of tempering that the already annealed steel. Annealing and quenching increase the hardness while the tempering decreases the hardness.**

1045 Steel Condition	Hardness Test 1 (HRB)	Hardness Test 2 (HRB)	Hardness Test 3 (HRB)	Hardness Test 4 (HRB)	Average Rockwell Hardness (HRB)
Initial Hardness	90.7	91.8	90.0	N/A	90.8
Austenitic State at 910°C	110.8	110.2	112.1	N/A	111.0
Tempering at 550 °C	98.6	99.3	99.0	99.6	99.1

As expected, the annealing process of the steels shows that there was an increase in hardness [4]. This is because quenching the steels to room temperature very quickly from the austenitic region creates a high degree of undercooling, so nucleation dominates the process as illustrated in Fig. 3. We end up with a microstructure that has lots of small fine particles (martensite) and a very high hardness because of its resistance to dislocation motion. This behavior is seen in both steel specimens through an increase in their respective hardness, with 1018 steel being is more robust than 1045 steel. 1018 steel is also affected less by heat treatments at the temperature range at which we tested. Table 4 shows that during the annealing and quenching process the 1018 steel's hardness increased by 6.2 % and a corresponding 2.1% decrease in hardness for the tempering process. The 1045 steel specimen's results indicated a 17.5 % increase in hardness during the annealing process and a corresponding 9.9% drop in hardness during the tempering process, Table 5.

For both steel specimens, we see that the tempering process is a tradeoff, where some hardness is lost but we gain back some ductility. For steel with a martensite microstructure, which is too brittle for practical use, tempering martensite allows the material to gain back some ductility while retaining hardness. This behavior is exactly what was observed in tempering the steels. While the hardness of both 1045 and 1018 steel decreased, they did not decrease to their initial value. Leading to the idea that the steels recover some ductility while retaining some of the hardness gained in the annealing and quenching process.

While 1045 steel is much more affected by heat treatments compared to 1018 steel. Both 1018 and 1045 steel are much more resistant to material processing than copper. As seen in Fig 4 and Fig. 5, copper is significantly affected by materials processing, specifically looking at coldworking and tempering of the specimen. While the steels were both tempered for 30 minutes and saw at the highest a 17.5 % increase in hardness (annealing and quenching the 1045 steel), copper in only one minute nearly halved its hardness value.

Conclusion

While ductile materials are more versatile for many applications, many times it is necessary to have a material that is both hard and ductile. To achieve this balance, materials can be strain hardened to increase hardness, and tempered or annealed to increase ductility. Copper's hardness increased dramatically with %CW, especially the first few %CW but it reached a point at which increasing %CW did not have much of an effect on the hardness of the copper. When tempering the copper we observed that much of the hardness reduction happens very quickly. One minute into the tempering process the hardness was almost halved. Steel can also be hardened by annealing and then rapidly quenching the steel. Thus, nucleation dominates over growth and the microstructure becomes dominated by lots of fine small particles known as martensite. Martensite is very hard but also very brittle. Martensite can be tempered to regain some of the ductility. The 1018 steel was more robust, meaning that it responded less to the heat treatments then the 1045.

References

[1] Callister, W. D., and Rethwisch, D. G., 2018, *Fundamentals of Materials Science and Engineering: An Integrated Approach*, 5th ed. John Wiley & Sons Inc., NJ, Chap 7.

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[6] Christopher A. Schuh and T. G. Nieh Schuh, C. A. and Nieh, T. G., 2002, "Hardness and Abrasion Resistance of Nanocrystalline Nickel," ADA417952, Materials Research Society, Boston, MA

[7] Callister, W. D., and Rethwisch, D. G., 2018, *Fundamentals of Materials Science and Engineering: An Integrated Approach*, 5th ed. John Wiley & Sons Inc., NJ, Chap 11.

Appendix A

Copper						
Initial Dimensions				Thickness (in)	Length (in)	Height (in)
				0.125	3.295	1.085
			Hardness Test 1 (HRF)	Hardness Test 2 (HRF)	Hardness Test 3 (HRF)	Hardness Test 4 (HRF)
Initial Hardness			71.5	75.2	77.4	74.7
Anneal Specimen for 15 mins @ 400°C then quench (~1s)			11.5	20.1	25.1	21.9
Relation between hardness and % thickness reduction (0% to ~60%)		Thickness	Hardness Test 1 (HRF)	Hardness Test 2 (HRF)	Hardness Test 3 (HRF)	Hardness Test 4 (HRF)
		0.125	20.1	25.1	21.9	N/A
		0.124	36.9	44.9	N/A	N/A
		0.122	53.9	52.9	53.3	N/A
		0.113	70	70.3	71	N/A
		0.106	75.4	74	76.2	N/A
		0.095	78.7	78.5	79.4	N/A
		0.0775	82.2	82.6	79.9	N/A
		0.063	85.3	84.2	84.1	N/A
		0.032	83	82.5	82.4	N/A
Temper thin specimen in furnace (1 - 20 mins) @ 400°C to find relation between tempering time and hardness	1 minute		42.4	41.3	42.8	N/A
	4 minutes		38.2	43.8	37.6	37.3
	8 minutes 20 seconds		38.5	39.1	39.5	N/A
	18 minutes 15 seconds		33.7	41.0	46.3	39.9

1018 Steel						
Initial Dimension				Thickness (in)	Length (in)	Height (in)
				0.120	1.705	1.705
			Hardness Test 1 (HRB)	Hardness Test 2 (HRB)	Hardness Test 3 (HRB)	Hardness Test 4 (HRB)
Initial Hardness			52	80.7	82.4	83.6

Hardness for quenching in water after at least 30 mins in furnace @ temp corresponding to austentic state (910°C)	84.8	90.2	87.7	N/A
Hardness for quenching again after 30 mins tempering @ 550°C of previously quenched specimens	82.9	85.2	85.6	86.5

1045 Steel				
Initial Dimension		Thickness (in)	Length (in)	Height (in)
		0.120	1.705	1.705
	Hardness Test 1 (HRB)	Hardness Test 2 (HRB)	Hardness Test 3 (HRB)	Hardness Test 4 (HRB)
Initial Hardness	90.7	91.8	90.0	N/A
Hardness for quenching in water after at least 30 mins in furnace @ temp corresponding to austentic state (910°C)	110.8	110.2	112.1	N/A
Hardness for quenching again after 30 mins tempering @ 550°C of previously quenched specimens	98.6	99.3	99.0	99.6