Behavior of Materials at High Temperatures;
Instilling a Healthy Uncertainty

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The events that occur in metallic materials heated to high temperatures determine material functionality, formability and fabricability. Few laboratory experiences allow engineers to explore the performance of real engineering materials at homologous temperatures greater than 0.8. Fewer still enable students to relate material performance at these temperatures to the microstructure of the material. Moreover, the relationship of material properties at lower temperatures are rarely graphically and directly connected to the conditioning of a material at higher temperatures. This paper describes the conception and execution of a laboratory experience to improve undergraduate students understanding of these complex material behaviors. In addition it includes portions which enable the student to quantify phenomena often discussed only in qualitative fashion. Laboratory procedure for the experiment is described in detail.

In this laboratory, students accomplish hot ductility testing using the Gleeble thermo-mechanical simulator, a system that has been successfully used to characterize the behavior and cracking susceptibility of numerous alloy systems. Students identify two key parameters of cracking sensitivity, the Nil Strength Temperature (NST) and the Nil Ductility Temperature (NDT) for alloy systems they test. Students pull samples to failure on heating to the NST and on cooling from the NST. Students record strength and ductility of each sample. This approach provides quantitative strength and ductility data under thermomechanical conditions which approximate industrial environments. Students examine fractured samples using scanning electron microscopy (SEM), optical microscopy (OM) and electron microprobe analysis (EMPA).

The laboratory allows students to observe changes in the mechanical properties of materials as a function of temperature. Students treat materials in rapidly changing environments which closely mimic those encountered in application, rather than in the mock contexts often discussed in the classroom or oversimplified cases typically treated in the laboratory. The laboratory discussed presents theory and application in a natural fashion, linked and mutually supportive. The paper discusses the exceptionally positive impact that this immediacy has an on student learning.

I. Introduction

The behavior of materials at elevated temperatures is a crucial but often ignored segment in the educational journey of many engineers. This is problematic as engineered systems will often be exposed to high homologous temperatures either during fabrication or when in service. Typically, curricular exposures to the high-temperature behavior of materials are limited to brief theoretical treatments in textbook readings or lecture. Laboratory experiences that treat elevated temperature

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behaviors are rare, rarer still are experiences which treat the steep spatial gradients and associated dynamic thermal histories in engineered materials and systems. A predisposition to quasi-equilibrium thermal conditions renders many laboratory exposures at best pedantic and at worst exercises that abrogate the fundamental purpose of laboratory-based learning experiences. Such treatments obfuscate critical information, and perpetuate erroneous mindsets as there are few, if any, processes or applications operating at elevated temperatures where thermal gradients are absent and where heating and cooling rates are not critical.

Well-developed and well-conceived laboratories are a key component in student learning, underpin subsequent independent project based learning and support the development of engineering judgment. Laboratories provide alternative means of developing understanding for the ever-increasing variety of learning styles represented in our classrooms. The contrast that laboratories provide to the classroom can reinvigorate students and provide them necessary vehicles to demonstrate the outcomes mandated in ABET's Engineering Criteria 2000. Therefore, even in difficult economic times laboratories can provide value far in excess of their cost.

Effective laboratories must be challenging and have verisimilitude to engineering practice. In creating the laboratory we must walk a very fine line. We must ensure that our students understand the goals of the laboratory, but not rob them of the joy of discovery. We cannot waste laboratory time, but must avoid being formulaic or plodding. Most of all, we must keep a freshness and a healthy uncertainty associated with the laboratory experience. Paradoxically, the ability to embrace uncertainty, and the willingness to make decisions when data is incomplete are key features in the make-up of successful engineers. Thus, teaching laboratories should hold the same enchantment and exhilaration for our students as research and applications laboratories hold for our graduates. Applied researchers go to the laboratory to coax truth from an inexpressive natural world, their aim is to sense, to evaluate, and, eventually, to progress. We must send our students to the instructional laboratory to accomplish these same goals.

Furthermore, instructional laboratories that stress the learning involved in doing enable faculty to pander to many different learning styles. They are customizable vectors to student perception. They provide real connections for students awash in the theoretical. They provide a safe transition across the convoluted borders that separate the odd and dissonant real from the uncluttered world of generalization described by precise equations and found in the answers to the even numbered problems in the back of textbooks. They are a necessary interlude where fundamentally self-absorbed, unsociable, covert egomaniacs discover the value of collective experience and collaboration, and develop skills in sharing and exchanging information. Laboratories, then, create a microcosm of, and a brief segue to, behaviors that are analogous to the stimulating milieu encountered in authentic occupational environments. They provide a prospect for legitimate eureka events that can rouse intellectual fires which can blaze for decades and which can afford illumination that reaches far beyond evident borders of the exercise. Laboratories that immerse students in significant tasks engender acceptance of ambiguity and apparent contradictions that place students on the path to the development of engineering judgment.

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Shared Responsibility / Shared Reward. One key to productive laboratories is the acknowledgment of each student's individual responsibility for group achievement. The effort required to accomplish laboratory goals should exceed the capacity of any single student, and require the sustained coordinated effort of the laboratory team. The instructor must develop an open learning environment and promote interdependence while fostering individual responsibility. As instructors, we can take a lesson from corporate America – rewards available to each lab group are based on group outcomes, individual rewards to group members are based on a collective assessment of each member by the instructor and by the group.

II. Desired Outcomes / Laboratory Objectives

In accordance with ABET outcomes oriented assessment, laboratory objectives are shared with students at the beginning of each laboratory, as are the instructor's desired outcomes. The course objectives are measurable goals that indicate how well the instructor's laboratory outcomes are achieved. Benjamin Bloom developed a taxonomy for categorizing the level of abstraction in questions that commonly occur in educational settings.(Bloom) Bloom listed six levels in his taxonomy. *Each laboratory* should provide the opportunity to exercise all of these cognitive levels. *Each student's* personal interaction with equipment/tools will lead to the accumulation of knowledge and skills required in the practice-oriented engineering profession.

The Instructor's Desired Outcomes

Many outcomes are universally associated with laboratories, and differ only in context. However, seven key outcomes, specific to this laboratory exist.

The first outcome is that students will learn how to use the Gleeble simulator, and be able to extrapolate to other potential uses for the test system.

A second outcome is that the students will appreciate the importance of thermal gradients in materials processing, and the impact they have on material behavior. The constant temperature properties of materials are just a starting point for processing or applying materials. The procedure of improving processes and applications requires far more information than is available in constant temperature tests. The behavior of materials in engineering applications can be understood only if this is appreciated.

A third outcome is that students will appreciate physical simulation. During the past three decades, industry has seen costs multiply many times. Where it was once practical to operate a process to study and optimize it, present day costs mandate the use of less lavish methods. The past three decades have also witnessed the greatest change in the ability to handle and control information ever experienced. Computer models of processes have proliferated, but basic information needed to improve the precision of the models is lacking. Conscience-less computer simulation provides great graphics but often other-worldly answers when not constrained by real-world limitations.

A fourth outcome is that students will appreciate that the effects of "minor" elements are not minor. Some elements, even at very low concentrations, can have dramatic impact on critical

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material properties. Apparently disproportionate affects are usually attributable to the concentration of the element at locations of singular importance in a material.

A fifth outcome is that students will understand the importance of surfaces in materials, and the gross differences between bulk and surface compositions. Surfaces are often locations of unique importance in a material. They are high-energy locations, preferred sites for nucleation events, and regions of disregistry where impurity atoms can accumulate. In fact, although major elements can be enriched or depleted at a grain boundary, it is primarily the enrichment of impurity atoms that gives rise to the greatest difference in composition as well as the most significant effects on properties.

A sixth outcome is that students will realize the importance of surface tension driven phenomena in solid materials. Surface tension is a particularly important feature in the behavior of materials because of the amount of surface area and the high surface energy associated with metallic materials. Surface energy is too often a neglected term in thermodynamic expressions for "free energy".

A seventh outcome is that students will be aware of the effects of rapid heating on the creation of liquid phases in otherwise solid systems. (Walsh) The abrupt appearance of liquid in a solid body can have drastic effects on mechanical properties, particularly when the liquid forms a continuous inter-granular film. Rapid heating can cause the formation of a liquid phase at temperatures far below that predicted by equilibrium treatments.

Laboratory Learning Objectives

By completing this laboratory participating students will demonstrate an ability to: 1. Apply the Gleeble simulator, quantitative microscopy and optical microscopy to make measurements of physical quantities, including testing and debugging an experimental system. 2. Devise an experimental approach, specify appropriate equipment and a set of procedures and implement those procedures. 3. Demonstrate the ability to collect, analyze, interpret data, and form and support conclusions. Make order of magnitude judgments about data correctness. 4. Identify the limitations of theoretical models as predictors of real world behaviors. Be able to evaluate whether theory adequately describes a physical event and establish and/or validate a relationship between data and underlying physical principles. Integrate thermodynamic and kinetic data. 5. Recognize unsuccessful outcomes and faulty construction or design, and modify the experimental approach accordingly. 6. Demonstrate appropriate levels of independent thought, creativity, and capability in problem solving in the real world. 7. Demonstrate competence in selection, modification, and operation of appropriate engineering tools and resources. 8. Recognize health, safety, and environmental issues related to technological processes and activities and deal with them responsibly. 9. Communicate effectively with a specific audience, both orally and in writing, ranging from executive summaries to comprehensive technical reports. 10. Demonstrate the ability to work in teams, including structuring individual and joint accountability, assigning roles and responsibilities, partitioning work, monitoring progress, meeting deliverable deadlines, and effectively integrating individual contributions into a final deliverable. (ABET)

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III. Equipment and Procedure - The Gleeble Simulator

The Gleeble is a fully computer interfaced device capable of simulating any thermal and/or mechanical history experienced by a material. The device employs a low frequency (60 cycle) alternating current to heat a specimen by resistance (impedance). The specimen is actually the secondary of a transformer in which the voltage is stepped down from 480V to 10V. The device switches 0.25 MW, so very high currents can pass through the sample. Specimen geometry is arbitrary, but is typically round bar or flat bar. Cross sections can be as great as 625 square mm, and heated lengths can be over 400mm. Specimen temperature is controlled by either thermocouples mounted directly to the sample or by an optical pyrometer. Pyrometer control is required for experiments involving carbon/carbon composites. The device can heat/cool at controlled rates from 10,000 C per second to 1 C per hour. Jaws that provide electrical contact grip the specimen. These jaws form the bed of a hydraulic mechanical test apparatus. Mechanical control is provided by any one of several modes; force, stroke, dilation. Thus the Gleeble allows the experimenter to control temperature and one mechanical variable while recording up to eight other signals. A transient data recorder incorporated into the control system gathers information. The Gleeble has been developed with both mechanical and thermal simulation capabilities; neither capability was developed in a secondary manner. The Gleeble is unique among simulators in that it performs thermal and mechanical tests equally well.

The equipment allows the study and test of materials in the same dynamic fashion they are fabricated and used. **The application of the Gleeble to any materials laboratory course is limited only by the exposure, resourcefulness and, occasionally, the courage of the user.**

IV. Materials

A number of different materials are used in this laboratory, each group is provided with twenty samples to test on-heating and on-cooling. Samples were supplied as 6.25mm diameter round bar stock, cut to 112mm lengths, with 12mm lengths threaded on each end. In any single class, three student groups are provided with identical samples and at least two different types of material are tested. The identical samples provide the capability for statistical basis in the experiment, the different types of materials allow students to compare and contrast behavior. Examples of systems tested in parallel include AISI 4130 and AISI 1030 (to examine the effects of alloying elements), annealed plain carbon steel and cold rolled plain carbon steel (to test the effects of prior grain structure), and AISI 304 and AISI 303 stainless steel (to test the effects of resulfurization.)

In this experiment, students are not told *a-priori* that they can share data to develop more meaningful conclusions and create a statistical base for inference! Students are asked to design a test matrix to examine high temperature ductility on-heating and on-cooling, based on the variables of material tested, peak temperature, jaw separation and specimen diameter. In discussion students determine that their goals can be achieved more rapidly, and more clearly by collaboration with the other groups, quite a discovery for the typical student. Samples are heated to a specific temperature, and pulled to failure. Students measure loads at failure, and the sample ductility. As part of the study, students characterize the microstructure of the material at room temperature, and at the series of test temperatures selected. Groups typically feel that they should

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repeat the test; because the initial consensus is that there was some sort of procedural or material problem with that particular sample. At this point, the real voyage of discovery has begun.

V. Theory

Heat affected zone (HAZ) liquation cracking is a type of high-temperature weld cracking which is found in a narrow region of the HAZ juxtaposed to the fusion boundary. This phenomenon is used as the context for the experiment because it is inherently interesting, and its study produces artifacts that capture the imagination of the student experimenter. Furthermore, the hot-ductility data is applicable to a wide range of other metallurgical process and service applications. HAZ cracking is encountered during the welding of many alloys, including commonly used steel, nickel-based materials and aluminum alloys. Though the phenomenon is not fully understood, the metallurgical basis for cracking hinges on the presence of a persistent liquid film at the grain boundaries in a material. The presence of a liquid film is a necessary but not a sufficient condition for cracking, the film must also exist during an interval when the thermally or mechanically induced strain exceeds its ability to accommodate distortion.

Though many techniques have been developed to quantify weldability and weld cracking sensitivity, the Gleeble thermomechanical simulator takes a unique approach to characterize this behavior. Many techniques create crack susceptible microstructures and then impose stress or strain to initiate cracking, measuring susceptibility in terms of number of flaws, length of flaws etc… In contrast, the Gleeble does not quantify cracking propensity by the extent of cracking, rather it fully characterizes the ductility of a material at high-temperature and relates this to cracking tendency. Thus, results are not specific to a welding operation.

In this experiment small, round tensile samples are fractured either during heating to or cooling from the nil-strength temperature. The nil-strength temperature (NST) is identified by heating a sample to high-temperature under a nominal load. The resultant fracture surface is invariably intergranular, and is created when the liquid layer surrounding the grain grows to a thickness which obviates capillary attraction, and allows the sample to separate under the slight applied load. The load required to fracture specimens on-heating and on-cooling is recorded, and the transverse reduction-in-area is determined as a measure of the ductility. Students then determine the nil-ductility temperature (NDT), the on-heating temperature where the ductility of the material essentially falls to zero. Students find the ductility recovery temperature (DRT), the temperature at which the material recovers an arbitrary perceptible ductility on cooling. Students plot the on-heating and the on-cooling ductility curves and interpret them.

Prior to testing, students are asked which experimental parameters are critical in the determination of hot ductility. Students hypothesize that heating rate, cooling rate, peak temperature for on-cooling tests, hold time at peak and test temperature, and stroke rate are key factors that can alter results in hot-ductility testing. Students are also asked what criteria could be used to assess weldability. Students typically suggest a number of measures, including many which have been used by investigators and reported in the open literature. These include a classification of ductility curve shape, the extent of the nil-ductility region, the rate of ductility recovery on-cooling, the extent of the NST-DRT or NDT-DRT, and the rates at which both

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ductility and strength are recovered in combination.

In their reports, students are asked to discuss the metallurgical phenomena and to formulate a scheme which explains hot-ductility and hot-strength data. Eventually they discuss three regions the first, where ductility decreases to moderate values as temperature is increased, a second where the ductility rises to very high values and a third where the ductility rapidly falls to zero. Over the same temperature range, the strength decreases monotonically, until a sharp discontinuity is seen in the third region. In the first region both softening and hardening processes occur during deformation, but work hardening dominates resulting in high strength but low ductility. At the beginning of the second region, softening processes begin to dominate over strain-hardening processes. Students observe that increased strain rates shift the start of region two to higher temperatures. The onset of this region is also observed to be a strong function of all the other material parameters which affect recrystallization. Finer grained materials appear to have higher ductility because of the increase in available recrystallization sites. This also helps explain some of the changes when materials are tested on-cooling. Samples heated to extremely high temperatures will have a greater grain size and also loose many of the grain boundary stabilizing precipitates found in ferrous and Ni base alloys. In addition, the general effect of alloying additions is observed to be increased strength and reduced ductility at temperature. Fractures in higher alloy materials take on an intergranular appearance because of the relative increase in strength of the matrix vis-à-vis the grain boundaries. Students discover that the onset of recrystallization is markedly dependent on the original microstructure. Students observe recrystallized grains in the microstructure of samples, but soon realize that they must determine if the recrystallization occurs after deformation, or during deformation by a dynamic and repeated recrystallization process. Very high ductilities can be achieved only by a dynamic process. Recrystallization increases ductility quickly, isolating flaws which begin to form at boundaries and triple points. Ductility increases up to the point where region 3 begins. At this point processes such as constitutional liquation and alloy segregation produce liquid in the microstructure at temperatures which can be substantially below the theoretical melting point of the alloy. Once liquid is formed, it wets migrating grain boundaries pinning and coating them. Zero ductility is observed when the boundaries are first completely coated by liquid, zero strength occurs when the boundaries are coated by a substantial thickness of liquid. The beginning of liquation can be correlated to an abrupt decrease in ductility and a discontinuity in strength. Students discover that even at complete wetting, substantial load is still required to overcome the attraction caused by surface tension, though measured ductility is zero. Given the very high surface tension associated with liquid metals, even with a boundary film only 60 angstroms thick the load required to cause fracture can be over 5 to 7 KSI!

Students are expected to correlate their mechanical results to the microstructures they observe using optical microscopy and scanning electron microscopy. Students are supplied with microprobe traces taken from samples similar to those they produce during the experiment. Students learn that OM and SEM can be complementary tools in analyzing fracture phenomena. Coupled with OM, SEM can clearly show inter-granular(IG), ductile rupture and mixed mode fracture surfaces. Furthermore, it allows the student to differentiate between liquation based IG fracture mechanisms and solid state based IG fracture mechanisms. Students are particularly

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impressed by ductile fractures produced when high-temperature IG separations formed by grain boundary sliding and/or separations initiated at triple points are blunted by dynamic recrystallization at the crack tip.

Students find that the hot-ductility test provides data which can be used to predict weld metal HAZ hot cracking. In general, alloys which have good high-temperature ductility when tested during heating, and rapidly recover ductility when cooled from the NST are crack resistant. Furthermore, strength values can be very helpful in identifying crack sensitive materials. Alloys with marginal ductility, but which have high strength at temperature are more resistant to cracking than similar alloys with low strength.

VI. Reporting

Students are required to keep a laboratory logbook, listing work done and observations made on each lab day. The lab book is signed and dated by all group members. Each group is asked to prepare a detailed formal laboratory report describing the experiment, providing data, discussing results and offering conclusions and suggestions for further study. The report must contain a one-page executive summary. In their report, the students are also asked to evaluate the laboratory and asked to suggest improvements. In addition to the verbal communication inherent in daily laboratory operation, groups are also asked to report their findings orally. During the oral presentation groups are asked questions about other alloy systems, and the effects of other process parameters that provide the students with "intellectual launch-pads" and mental "room to roam".

VII. Evaluation

Students are evaluated by group and as individuals. Rewards (grades) are provided based on a corporate model. The instructor evaluates groups; rewards (points) available to each lab group are based on group outcomes, such as the quality of the report and presentation. The instructor bases individual rewards (grades) to group members on a collective assessment of each member by other members of the group; however, the total points available to the group delimit rewards.

VIII. Conclusion

Student experience with the laboratory has been very positive. Comments indicate that students are interested in the material and energized by it. The opportunity for genuine discovery, even though "engineered" into the laboratory, is considered a strong vehicle to help students develop true professionalism, even while cloistered in the academic setting.

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