A High Temperature Hardness Testing System for Mechanical Characterization of Engineering Components

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Abstract

Mechanical properties of materials at high temperatures often show a significant departure from room-temperature behavior. In the study of materials science, an understanding of mechanical behavior as a function of temperature is quite valuable. One of the elegant ways to determine tensile strength of metallic alloys is to measure the material hardness, in which the hardness acts as a "strength microprobe."[1]

With this aim in mind, we put together a high-temperature microindentation hardness tester by assembling various mechanical components and electrical systems for operation at high temperatures in the range of room temperature to about 1000°C. The basic hardware components were from a commercial hot-hardness tester which was no longer functional. These components were acquired from a commercial cutting tool manufacturer for a student project. In this paper, we describe key aspects of the system operation and illustrate the application using hardness data obtained on a series of cutting tool materials at different temperatures.

The unique aspect of this capability for the University is that it is the only hot-hardness testing system available in the state of Arkansas, and provides an excellent opportunity for classroom education and research on high temperature mechanical behavior of cutting tools, steels for die casting dies, tool steels, engine and turbine parts, ceramics and composites operating at elevated temperatures. It is available as a valuable educational and research tool for students and faculty, as well as to regional manufacturing industries, who may be able to use this testing service, thereby supporting the operation and training opportunities for students on an on-going basis.

Introduction

The discovery of materials and their unique properties has played a vital role in the evolution of human civilization, from the Stone Age to the present day, as depicted in Fig. 1[2]. As new discoveries are made, and new materials are developed, their properties and ability to manipulate these properties have fascinated the scientists and engineers for centuries. In various industries and applications, the range of materials available today demonstrates the genius of human mind, to synthesize, manipulate, predict and use materials and their unique properties, in order to enhance performance of components and structures, push the limits of capabilities of materials, and discover new phenomena that lead to even more unique materials.

One of the most common features of material use is the inevitability of material failure, either due to wear, chemical corrosion or other factors. The durability of materials depends, in many
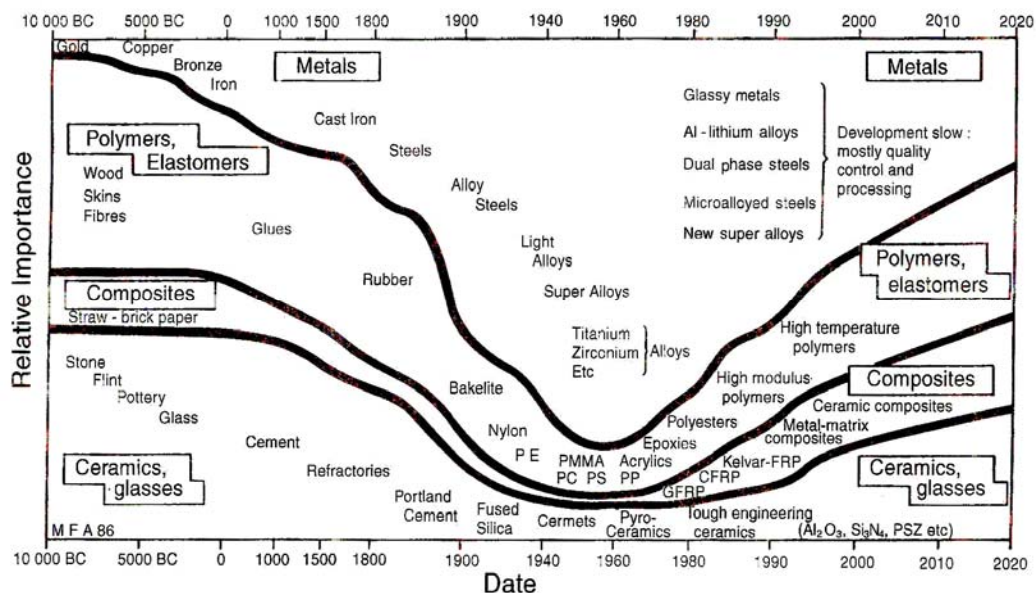

Fig. 1: The evolution of materials through ages (from Ashby, Materials Selection in Mechanical Design, Third Edition, Elsevier 2005.)

cases, on their strength and resistance to environmental factors. Materials that are found to be strong, hard and durable at ordinary temperatures often show degradation in their properties at elevated temperatures. Thus, it has always been of paramount importance to understand the behavior of materials at high temperatures, typical of many service environments, e.g. high temperature ovens, automotive engines, power plant equipment, industrial gas turbines, jet engines and so on. Failure of materials at high temperatures can occur due to oxidation, corrosion or applied stress, often over a period of time. Time-dependent deformation of a material when subjected to a constant load or stress is called creep, which is accelerated at high temperatures. Other modes of high temperature failure involve erosion, abrasion and fatigue, and often a combination of these factors. An understanding of the properties of materials at high temperatures, therefore, is an important aspect of learning about materials.

One of the most common properties of materials that most people are familiar with is strength. The strength of a material is typically conceived of as resistance to deformation, resistance to fracture, or simply "durability." Often, the toughness of a material is thought of as an indicator of its durability. The most common method for measuring this property is the ubiquitous tensile test in the case of metallic materials, and compressive or flexure test for ceramics and other types of brittle materials. Another most common method is to measure the "hardness" of a material. Hardness is defined as resistance to penetration by another object. It is measured by a number of different techniques, such as Brinell, Vickers, and Rockwell test methods[3], in which a hard indenter (typically a pyramid shaped diamond tip or a spherical hardened steel or cemented carbide ball) is pressed into the surface of the specimen under a specified load. The applied load, divided by the surface area of the indentation, gives the hardness value, which is typically expressed in the same dimensional units as the strength, e.g. MN/m^2 (MPa). In the case of

Rockwell test, the hardness is expressed as an arbitrary scaled number that depends on the depth of penetration of the specific indenter under a specific load.

The strength of a material is related closely to its atomic structure, bonding between the atoms comprising the material, any processing conditions, and the so-called microstructure. Studies have shown that there is a distinct correlation between the strength of a metal and the size of the "grains" or crystals that make up the metal in the bulk form. This relationship is called the Hall-Petch relationship, and can be described as:

$$\sigma = \sigma_o + \frac{k}{\sqrt{d}} \tag{1}$$

where σ is the strength (or resistance to permanent deformation), σ_o and k are constants for a particular material, and d is the grain size. In the case of many metals and alloys, there is an empirical correlation between strength and hardness. Thus, the Hall-Petch relationship also applies to hardness, and one can write:

$$H = H_o + \frac{k}{\sqrt{d}} \tag{2}$$

where H is the hardness of the material, and other terms have the same meaning as before. Thus, it is often possible to estimate the tensile strength of a material by measuring its hardness, which is a simpler and faster test method as compared to the tensile test. Thus, the hardness acts as a "strength microprobe."

High Temperature Behavior

Hardness, strength and other mechanical properties play a significant role in the design of materials for various structural applications. These properties often combine interactively to
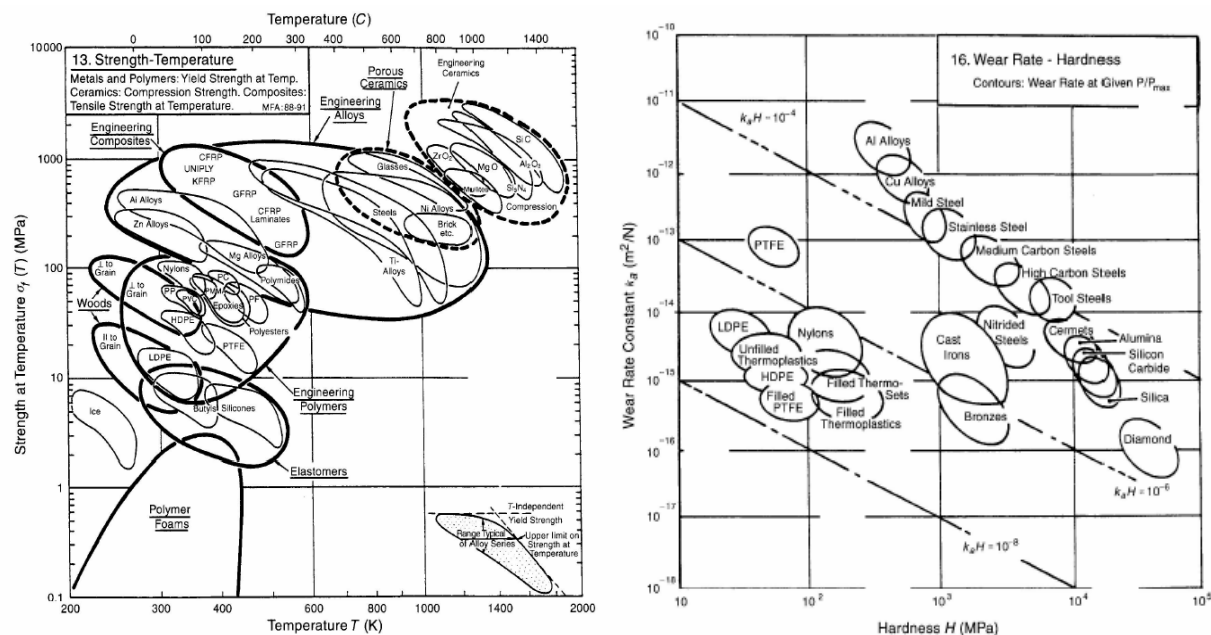

Fig. 2: Mechanical property maps for engineering materials: Left – strength versus temperature, Right – wear rate versus hardness (From Ashby, Materials Selection in Mechanical Design, Third Edition, Elsevier 2005.)

provide usable guidelines for the proper selection of materials for given applications. Ashby[2] has constructed a series of property "maps" that depict these relationships between materials properties, for a wide range of materials, as shown in Fig. 2. As one would expect, the strength and hardness of materials decrease as the temperature increases. The near-horizontal part of each lozenge in the diagram at left shows the strength in the regime in which temperature has little effect; the downward-sloping part shows the more precipitous drop as the maximum service temperature is reached. In other words, as the temperature increases, the strength may drop at a faster rate than at a lower temperature. Therefore, in many applications where high temperatures are involved, it is critical to know the properties at the service temperatures. The measurement of strength at elevated temperature is tedious, expensive and quite time-consuming. On the other hand, it is relatively easier and faster to measure the hardness of materials at elevated temperatures, if a suitable apparatus and method are available. Since a much smaller sample is needed for hardness test, it is relatively cheaper and easier to prepare test specimens.

Implementation of a Hot Hardness Test Method for Materials Laboratory

We have implemented a unique high-temperature micro-indentation hardness tester in the Surface Engineering Laboratory at the University of Arkansas, by assembling various mechanical components and electrical systems for the operation at high temperatures in the range of room temperature to about $1200^{o}C$. The basic hardware components were obtained from a commercial company which had dismantled and discarded their equipment since it was no longer functional. The equipment was assembled from various components and modules as a graduate student project, and tested to verify the operation of different functional modules of the system, such as the heating system, the vacuum and water-cooling system, the loading system and the measurement system.

Fig. 3: Nikon QM Hot Microhardness Tester at the Surface Engineering Laboratory, University of Arkansas

The unique aspect of this capability for the University is that it is the only hot-hardness testing system available in the state of Arkansas. It provides an excellent opportunity for classroom education and research on high temperature mechanical behavior of cutting tools, steels for die casting dies, tool steels, engine and turbine parts, ceramics and composites operating at elevated temperatures. The equipment is now available as a valuable educational and research tool for students and faculty, as well as to regional manufacturing industries, who may be able to use this testing service, thereby supporting the operation and training opportunities for students in an on-going basis. The equipment can be used as a complement to a graduate level course on advanced materials, physical metallurgy or materials in design. At the undergraduate level, a laboratory module may be developed for demonstrating the effect of temperature on the hot hardness of a heat-treated carbon steel and a tool steel, to illustrate the effect of alloying elements in tool steel.

QM Hardness Tester

The QM high temperature hardness tester permits observation and micro-hardness measurement of a variety of materials, such as metals, alloys, ceramics, composites and even coatings at any temperature ranging from room temperature to about 1200°C. The basic design of the instrument and its various modules can be seen in Fig. 3[4]. The various modules are shown by arrows, and comprise a well-integrated, high-precision measurement system for the hardness of materials over a wide range of temperatures.

The entire equipment is divided into four modules. These include the power source and vacuum control module that controls main power source and vacuum in the test chamber. The valve control module carries all the valves that need to be closed or opened while developing the vacuum in the chamber. Loading control module contains all the switches that activate the indentation process. There is a facility for setting the indentation time in this module. The heating control module maintains the desired temperature by toggling with the solenoid switch which switches OFF the heating process when desired temperature is reached and turns it back to ON when the temperature falls below the set temperature. The console desk carries the vacuum chamber in which the specimen heating furnace and specimen holder are placed as shown in Fig.4. The indenter heating furnace and indenter holder are placed in the lid of the vacuum chamber, shown in Fig. 5.
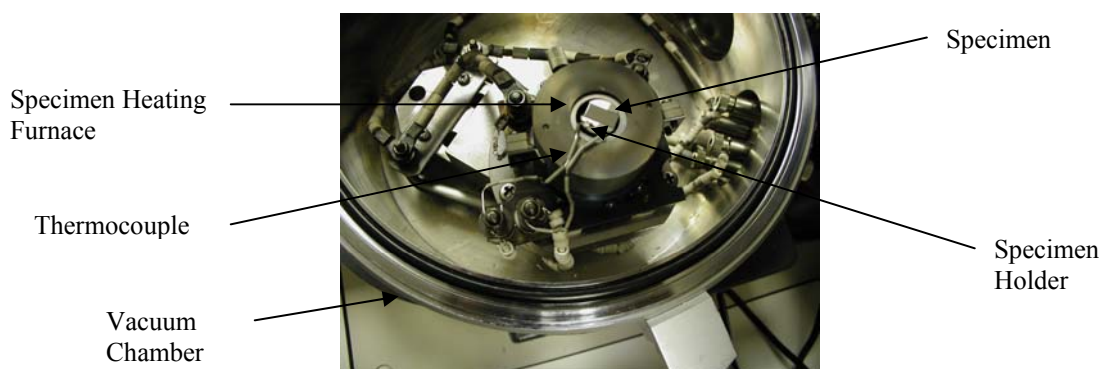

Fig. 4: Specimen heating furnace and specimen holder

The rotary pump connected to the main unit pumps the air out of the main chamber in order to develop vacuum. The equipment needs to be kept cool at all times to maintain the vacuum pressure. For this purpose, cold water is constantly circulated throughout the machine with the help of a water-cooling system. The water cooler and rotary pump are shown in Fig. 6. The temperature control unit shown is Fig. 3 is used to set the desired temperature at which the hardness measurements are to be carried out. This unit continuously monitors the change in temperature.
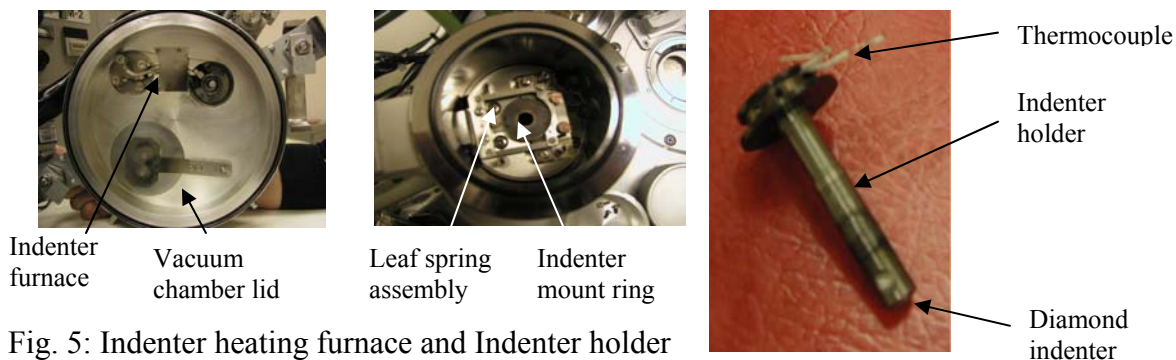

Fig. 5: Indenter heating furnace and Indenter holder

The required vacuum inside the chamber should be at least 10^{-4} Torr. After the test chamber has reached the desired vacuum, the heating units are turned on to achieve the desired temperature for the indenter and the specimen. The indenter assembly must be heated and controlled separately during testing. If the indenter is not heated, it can act as a heat sink when it touches the sample surface, causing fluctuations in temperature during measurement. The indenter presses into the surface of the specimen for the set time and then returns to its original position. The rate of indenter load application is preset to the desired value. At the completion of the loading cycle, the specimen assembly moves back to the initial position under the observation window. Two types of specimens can be used, both of which are made to precise dimensions in order to fit exactly into the specimen holders. The rectangular specimen is 5 mm × 5 mm × 10 mm, and the cylindrical specimen is 7 mm dia. × 7 mm long. The top surface of the specimen is lap-finished to make it easy for observation under the microscope.

Fig. 6: Left - Water cooler, and Right - Rotary Pump

The measuring microscope is brought into position to measure the diagonals of the indentation. The system is calibrated to give the length of the diagonals in microns at a magnification of

400X. The hardness can be obtained from the conversion tables, or calculated using the formula for Vickers hardness, which is:

$$HV = 1854.4P/d^2 \qquad (3)$$

where P is the applied load in grams and d is the average indentation diagonal in microns. The hardness of materials can be measured under vacuum or in an inert atmosphere such as argon.

Operational Aspects of Hot Hardness Tester

The equipment is easy to use and the sample preparation is simple. The main cost of the test method is the cost of diamond indenter. At present, a new Vickers diamond indenter costs about $750, and a refurbished one costs about $400. This is the main consumable cost, other than the high-purity argon required for initial purging of the loading chamber. Other periodic costs include diffusion–pump oil, which may have to be replaced at some point depending on usage, and supplies for metallographic sample preparation for polishing the specimens. If used carefully, it should not be necessary to replace other working components frequently. These include the specimen furnace, indenter furnace and leaf-spring assembly for loading. The latter is the most critical component of the system, and requires careful handling. If the leaf-spring is damaged, the instrument is unusable. Current replacement costs for these items are about $500 for indenter furnace, $1,000 for specimen furnace and about $2,000 for the leaf-spring assembly.

It is worth noting at this point that, the Nikon QM Hot Hardness Tester is a semi-automatic apparatus, which is no longer manufactured. The fully automatic system (QM-2) costs approximately $200,000 to $250,000, but may not be available at this point, since Nikon has announced recently that they will be discontinuing the sales and support for this equipment.

Measurement of High Temperature Hardness of Cutting Tool Materials

Tests were conducted on carbide cutting tool samples The purpose of these tests was to test the operation of the equipment, and to validate the results based on prior data reported by Valenite, Inc., the previous owner (and donor) of the equipment. These tasks were completed during the calibration and testing of the system functionality, and results verified using standard test blocks of calibrated hardness values.

Table. 1 Hardness of cemented carbide cutting tool grades from room temperature to 800°C.

Sample Composition	WC + 6% Co	WC + 13% Co	WC + 15.5% Co	WC +25% Co
Temperature, °C	Sample A	Sample B	Sample C	Sample D
Room Temp	1103	1003	917	740
200	1030	883	698	638
400	900	793	642	466
600	788	671	560	343
800	563	431	336	209

Next, a series of cemented carbide test samples of different compositions were obtained from Kennametal, Inc., Rogers, AR. The samples were cut to the required dimensions and the top face

was lap-finished in order to be able to see through the microscope. Vickers hardness tests were conducted on the samples with a 500g load at temperatures ranging from room temperature to 800°C. A minimum of three readings were taken for each data point, and averaged. Table 1 and Fig. 7 show the hardness values for different samples at different temperatures.

Discussion of Results:

We note two specific trends in these results. As stated at the beginning, the hardness (and strength) of any material decreases at higher temperatures, due to the softening of interatomic bonds. When a sample is composed of constituents with different melting points, as is the case with cemented carbides, there can be a significant change in hardness as the temperature is increased, even though some of the constituents may be better able to retain their hardness at high temperatures. Different grades of carbide tools experience different degrees of softening at high temperature, primarily as a result of their chemical compositions. Cobalt (melting point
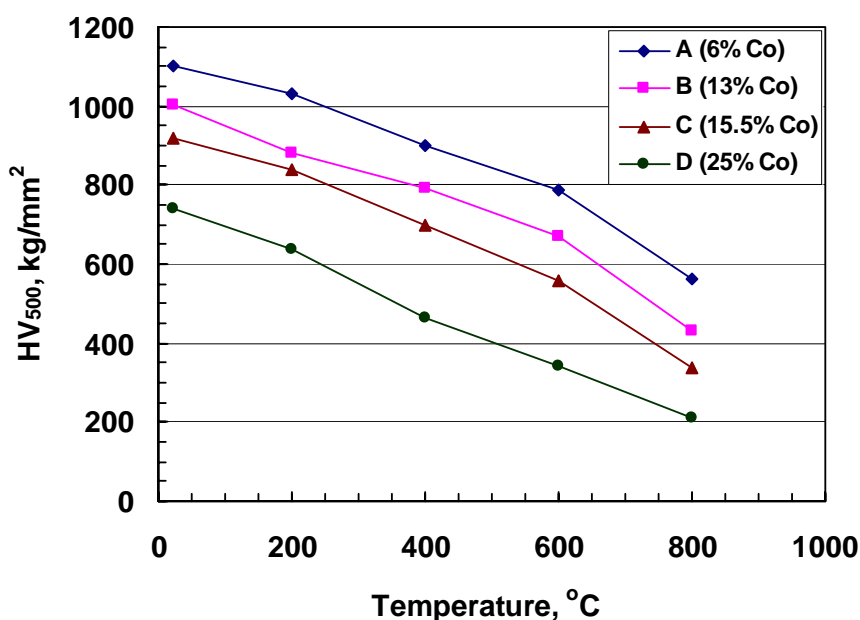

Fig. 7: Effect of temperature and binder content on the hot hardness of cemented carbide cutting tools

1495°C) acts as a binder for the hard tungsten carbide (WC) grains (melting point 2780°C), "cementing" them together into a strong and hard composite alloy. Thus, the higher the amount of cobalt (which is much softer than WC), lower is the overall hardness. When the tool is heated to a high temperature, as for example during machining, the cutting edge can become softer, and deform if the tool pressure at the cutting point is high. This leads to a rapid wear of the tool. Also, it is known that the metal being cut can react chemically with the tool material and cause "chemical" wear of the cutting tip. Therefore, it is extremely helpful to know the high temperature behavior of these tools under actual service conditions.

Studies of hot hardness of fine-grained ceramics have shown that it is possible to elucidate the mechanism(s) by which the material deforms or softens at elevated temperatures. In a study of hot indentation of yttria-stabilized tetragonal zirconia (YSTZ), Prabhu and Bourell[5,6] showed that

the hardness dependence of the ceramic changed drastically around 1100^oC due to a change in the dominant deformation mechanism. Hsu[7] used hot hardness to correlate elevated temperature tensile properties of a ferritic steel, and Nieh and Wadsworth[8] studied the high temperature deformation behavior of refractory metal beryllides used for advanced aerospace structural
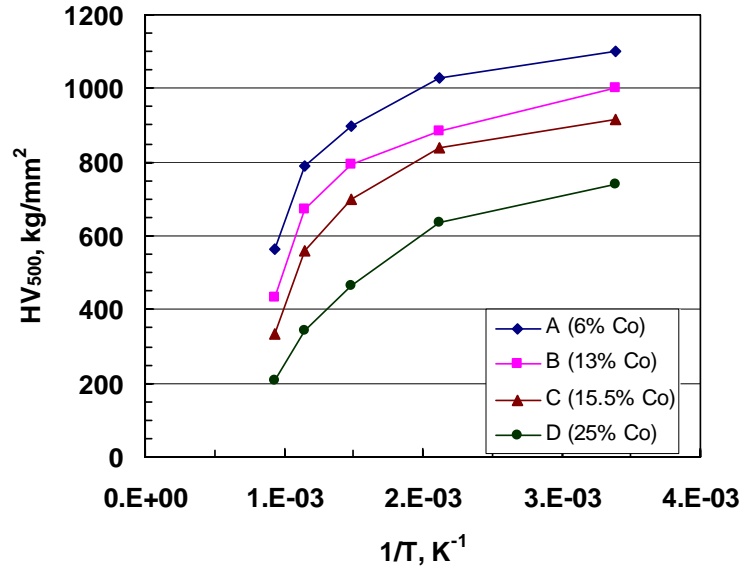

Fig. 8: Plot of Vickers hardness against inverse of absolute temperature. The change in the slope of the lines suggests a change in the dominant deformation mechanism as temperature is increased

applications. These studies demonstrated that the hot microindentation technique is a very sensitive method to detect and define the mechanisms of deformation of structural materials at high temperatures. The present data, plotted as shown in Fig. 8, illustrates this trend for the cemented carbide samples as well.

Deformation of materials at elevated temperatures is often described by a phenomenological equation of the form:

$$\dot{\varepsilon} = A\sigma^n \cdot e^{(-Q/RT)} \tag{4}$$

where $\dot{\varepsilon}$ is the strain rate, σ is the flow stress, Q is the activation energy for deformation, T is the absolute temperature, R is the universal gas constant, and A and n are material constants[9]. Since the rate of loading $\dot{\varepsilon}$ of the indenter in a hardness test is preset to a constant value, and considering the proportionality between hardness and applied stress, Eq. 4 may be modified as:

$$\left(HV\right)^n \propto Ae^{(Q_H/RT)} \tag{5}$$

where Q_H may be called the activation energy for deformation due to indentation, and can be determined from the slope of the hardness versus 1/T plot[5]. In the present work (Fig. 8), it is necessary to obtain more data to precisely define the slopes of the various curves, and to normalize the data in order to combine the effects of different parameters, before a definitive determination of activation energy, and deformation mechanism, can be made. This work is in progress.

Summary and Conclusions

The paper describes the setting up of a versatile hardness testing technique and apparatus for laboratory studies of hardness of materials. This tool is a valuable new addition for providing students at the undergraduate and graduate levels an insight into the inter-relationship between the strength and hardness of materials, and the effects of various materials and processing parameters on the mechanical properties. Results of hardness measurements at different temperatures on cemented carbide cutting tool materials show that the hardness is a function of temperature as well as composition of the tools. The test data can be used to elucidate deformation mechanisms of the tool materials at different temperatures. This information is valuable in understanding the wear and failure of cutting tools during aggressive machining conditions.

Acknowledgments

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