Vickers Microindentation Hardness Testing of Brazed Joints in Aluminum

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ABSTRACT: A procedure is described for performing Vickers (diamond pyramid) microindentation hardness profiling across four brazed joints in a previously fabricated and metallographically-prepared aluminum section to determine the hardness of the base metals and of the joints. Analysis of the measurements was performed to understand the variation in hardness that occurred during the creation of the brazed joints and related to estimates of tensile strength using a tensile strength-hardness correlation available in the literature. Data analysis was accomplished using a spreadsheet and its plotting capability. Subsequent to the hardness testing, low magnification digital images of the indented surface were obtained and used in the analysis and as part of the presentation of results. As an **optional** activity, the Cambridge Engineering Selector software can be used to generate a plot showing the correlation between tensile strength and hardness for various families of materials.

KEY WORDS: Vickers microindentation hardness, brazing and brazed joints, aluminum alloys, mechanical property correlations, Cambridge Engineering Selector software

PREREQUISITE KNOWLEDGE: junior/senior-level undergraduate laboratory experiment requiring knowledge of mechanical properties and phase diagrams and transformations as described in an introductory materials science course and accompanying laboratory course together with follow-on courses on these materials science topics. *(Instructor Note 1)*

OBJECTIVES:

(a) Experimental Goals:

1. To perform a calibration of a Vickers microindentation hardness tester using a standard test block;
2. To measure the Vickers microindentation hardness profile across a set of brazed joints in an aluminum alloy; and
3. **(Optional)** To obtain a computer-based mechanical property correlation.

(b) Learning Goals:

1. To be able to perform Vickers microindentation hardness testing, a prominent technique for characterizing the mechanical response of materials, and to analyze the resultant hardness values;
2. To be able to describe brazing, a technology widely used to join metals and alloys, and to identify its major industrial benefits and applications; and
3. **(Optional)** To be able to create a computer-based mechanical property correlation for various materials.

TYPE OF MODULE: Laboratory experiment

TIME REQUIRED: Hardness testing takes two to three hours; see *Instructor Note #1*.

MODULE LEVEL: Advanced undergraduate

MatEd CORE COMPETENCIES COVERED:

- 0.B Prepare tests and analyze data
- 1.A Carry out measurement of physical properties
- 6.A Apply basic concepts of mechanics
- 8.A Demonstrate the planning and execution of materials experiments
- 8.C Perform visual and nondestructive testing methods
- 9.C Distinguish processing methods for aluminum and aluminum alloys
- 16.A Distinguish effects of processing and manufacturing variations on material properties

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EQUIPMENT AND MATERIALS: (1) LECO microindentation hardness tester (model M-400) equipped with a metric micrometer x-y translation stage and a Vickers indenter; (2) LECO universal clamp and leveling device (LECO part no. 862-690); (3) Vickers test standard (LECO block no. 58-558; VHN = 710.5 kg_f/mm^2 for 300 g_f indenter load); (4) aluminum alloy sample containing brazed joints; (5) Wilson (Instron) Desk Chart 60 (Hardness Conversion); (6) Cambridge Engineering Selector EduPack software (2012).

SAFETY PRECAUTIONS: No particular safety precautions are necessary. However, care must be taken to avoid moving the sample once the hardness tester commences its operating cycle.

INTRODUCTION:

General Background: Indentation hardness testing [1] involves applying a constant load, P, to an indenter having any one of a variety of common shapes, such as a ball, cone, or pyramid (Refer to Table 6.9, Shackelford [2].). For Brinell, Vickers, and Knoop hardness tests [3,4], the resulting diameter or some characteristic dimension of the residual (plastic) impression formed in the surface of the material being tested is optically measured after the indenter has been applied and removed. This measurement allows determination of either the contact area, A_c, or projected area, A_p. The hardness pressure is then computed by dividing P by either A_c or A_p depending on the test being performed (Again refer to Table 6.9, Ref. [2].).

Current Work: Vickers (diamond pyramid) microindentation hardness testing [3,4] is a particularly effective “strength probe” [5] in characterizing the material in and around phase boundaries, such as those present in braze or weld joints [6]. Once the indenter and sample are properly aligned and the tester has been calibrated, hardness testing is easily performed, making it highly useful for obtaining such hardness profiles. This approach has been used in steel carburization [7,8] and case hardened [9] depth studies and in inhomogeneous deformation characterizations [10,11] after metals/alloys underwent bulk-deformation processing.

Vickers hardness numbers (VHN’s) are calculated using

$$\mathrm{VHN} = 1.8544\ P/d^2, \qquad (1)$$

where P has units of kg, and d is the hardness impression diagonal length, mm.

The purpose of the first portion of this experiment is to measure the Vickers microindentation hardness across a number of brazed joints in an aluminum alloy [12]. *(Instructor Note 2)* Brazing is the joining of two base metals via a filler metal.[13] While there are many options for joining metals, brazing has several advantages in forming permanent, strong joints. Brazing operations apply heat broadly to the base metals. The filler metal is then placed into contact with the base metals and melts. The filler subsequently wicks into the joint because of capillary action. When clad brazing sheet is used, the filler metal is already available where all joints need to be formed. Importantly, the base metals in brazing processes are not actually melted, compared to what occurs in welding. This results in lower processing temperatures for brazing with lower energy requirements.

The vacuum-brazed sample (Figure 1) characterized in this study was provided by ALCOA Mill Products in Lancaster, Pennsylvania, and involved modified 3003-H24 aluminum (Al with controlled Cu and Mn additions) [14] core sheet clad on both sides with 4004 aluminum (Al/10 wt% Si/1.2 wt% Mg) brazing alloy to create the brazed joints. The sample also contained a dilute 6000-series aluminum (Al with Mg and Si) extrusion separating the two brazed joint assemblies.

As an **optional** follow-on activity, a material property correlation for tensile strength vs. Vickers hardness was obtained using the Cambridge Engineering Selector (CES) software. *(Instructor Note 3)* This exercise is both feasible and worthwhile because hardness offers a measure of a material's resistance to penetration that is dependent on such mechanical properties as tensile strength and strain capacity.

PROCEDURE:

A. EXPERIMENTAL

1. Vickers microindentation hardness testing (An alternate exercise is provided in *Instructor Note 4*.)

Before commencing sample testing, it is necessary to use a calibration block of known hardness to verify that the tester is working properly. *(Instructor Note 5)* The objective of this work was to perform Vickers microindentation hardness testing across two brazed joint assemblies in a modified 3003 aluminum core sheet sample using a LECO M-400 tester. *(Instructor Note 6)*

Record measurements and any relevant observations in a laboratory notebook with appropriate drawings.

(1) Perform a calibration measurement using a load of 300 g_f for 25 s dwell applied on a standard test block placed in a universal clamp and leveling device to ensure that the surface to be indented is orthogonal to the indenter axis. If the measurement lies outside the specified range (In this case, VHN = 710.5 ± 3.9 kg/mm^2.), repeat the measurement since properly seating the block can be an issue.

(2) Using an applied load of 10 g_f for 25 s dwell, obtain a series of twenty hardness measurements on the sample over a 5.875 mm distance perpendicular to four brazed joints allowing them to be profiled. This is accomplished by manipulating the translation stage x-axis micrometer.

2. **(Optional)** Computer-based mechanical property correlation

Among its many capabilities, the CES software can easily provide linear or logarithmic plots of property A (appearing on y-axis) versus property B (appearing on x-axis) for a variety of metallic and non-metallic materials.[15] With such plots, it is possible to establish many interesting material property correlations. How property A varies with property B is easily visualized for all of the materials included in the software's database, and where specific materials lie on the plot can be conveniently identified and annotated. *(Instructor Note 7)*

Print a plot for taping into your notebook and turn in a copy with the laboratory reporting exercise.

(1) Prepare a logarithmic plot using the CES software **operating at Level I** for tensile strength versus Vickers hardness.

(2) For this plot, locate and label the following materials:

(a) alumina;
(b) aluminum alloys;
(c) bamboo;
(d) cellulose polymers (CA);
(e) CFRP, epoxy matrix (isotropic);
(f) flexible polymer foam (MD);
(g) silica glass;
(h) stainless steel; and
(i) stone.

B. ANALYSIS

Perform the following analyses and respond to any questions as completely as possible being sure to show all of your work and reasoning as partial credit can be earned.

1. Vickers microindentation hardness testing

a. Compute the VHN for each hardness impression employing Equation (1). Using Excel (or equivalent), create a properly labeled linear plot of VHN versus distance from the sample edge. Discuss the trend that exists.

b. Using Excel (or equivalent), create a properly labeled linear plot of tensile strength, ksi, versus Knoop hardness number (KHN) obtained at 500 g_f for most non-ferrous metals as given in Wilson (Instron) Desk Chart 60 [16]. Obtain the trend line equation and discuss the trend that exists.

c. Using Excel or equivalent, create a table giving the impression designation (e.g., number), VHN, estimated tensile strength (using the equation for the trend line in the second plot), and description of the impression location in the sample (e.g., near brazed joint, in brazed joint, or center of clad brazing sheet). Compare strength estimates for the center of the modified 3003 aluminum core sheet with corresponding literature values (e.g., in Ref. [17]).

2. **(Optional)** Computer-based mechanical property correlation

   a. Referring to your CES plot of tensile strength vs. Vickers hardness, discuss any trend that exists.

COMMENTS with Sample Data and Plots:

A complete set of the experimental measurements was obtained twice to verify the results. The data appearing in this section are considered to be representative.

<u>Vickers hardness testing:</u> A plot of Vickers microindentation hardness vs. distance from one external edge of the sample is given in Figure 2. Most prominent were two peak-valley profiles associated with the two brazed joint assemblies appearing in the photomicrographs in Figures 3 and 4. The highest hardness values were found for impressions numbered three, seven, twelve, and sixteen that were put in the brazed joints where melting and re-solidification of the braze liners had occurred to form a metallurgical bond between the extrusions and the braze sheets [18,19]. These high numbers are attributed to solid solution strengthening and some precipitation hardening that occurred in the joint; the pro-eutectic aluminum grains in the solidification zone trapped Si and some Mg in solid solution which provided hardening through solute strengthening and through precipitation hardening that developed during post-brazing natural aging.

Elevated hardness values were also found in the regions of the modified 3003 aluminum core sheets closest to the brazed joints for impressions numbered four, six, thirteen, and fifteen. These regions are darker than the two centers indicating that Si and Mg from the brazing alloy had diffused into both surfaces of the sheets causing solution hardening (and hence higher hardness) to occur. The lowest values (for impressions numbered five and fourteen) were at the nominal centers of the modified 3003 core sheets, being significantly lower than the dilute 6000-series aluminum extrusion.

Table 1 provides the relationship between distance from the sample edge, Vickers hardness number, and corresponding <u>estimated</u> tensile strength, along with identification of the physical location of the hardness impressions. The tensile strength <u>estimates</u> were obtained using the relationship between tensile strength and Knoop microindentation hardness (500 g_f load) appearing in Figure 5. *(Instructor Note 8)* The estimated tensile strengths for the centers of the two cores of the modified 3003 aluminum sheets were 14.4 and 22.2 ksi (average = 18.3 ksi) and compare rather favorably with reference values of 16.0 and 23.0 ksi for 3003-O and 3003-H22 aluminum, respectively [17]. *(Instructor Note 9)*

In summary, the presence of the four brazed joints was easily distinguished from surrounding base metal since each joint had the highest hardness value relative to the hardness values of the surrounding materials. The overall hardness difference averaged 55.2 and 32.4 kg_f/mm^2 for the left- and right-hand joint assemblies, respectively. The highest hardness values in the brazed joints were attributed to a combination of solid solution and precipitation hardening of the filler metal which contains both Si and Mg. A secondary hardening effect occurred in the regions of the modified 3003-O cores of the aluminum brazing sheets immediately adjacent to the brazed joints where solution hardening also occurred because Si and Mg in the brazing alloy diffused into these regions. Diffusion of these elements did not extend into the sheet centers where estimates for tensile strength for the modified 3003 cores, obtained from a published tensile strength-hardness correlation, compared reasonably well with available literature values.

Uncertainty analysis/source of error: The readability of the micrometer eyepiece on the hardness tester was 0.0005 mm. The largest source of error in this experiment occurred in measuring the diagonal lengths of the impressions. Each of four students made a length measurement, and the spread ranged from 0.0001 mm (corresponding to ΔVHN = 1.2 kg_f/mm^2) to 0.0135 mm (corresponding to ΔVHN = 12.0 kg_f/mm^2); the average spread in diagonal lengths for the 20 impressions was 0.0005 mm.

Computer-based mechanical property correlation: The logarithmic plot of tensile strength vs. Vickers hardness in Figure 6 shows a strong correlation with strength increasing as hardness increases. Since tensile strength is defined as the highest measured load divided by the initial load bearing area and typically occurs with considerable plastic deformation, it is not surprising that the two properties relate so well because Vickers hardness gives a measure of a material's ability to deform plastically in accommodating the indenter.

INSTRUCTOR NOTES:

1. The hardness testing described in this experiment can be accomplished during three (3) one-hour periods held on consecutive class days. Analysis and interpretation of hardness results were performed outside of class, although there was sufficient time in lab for some discussion. Students gained familiarity with using the CES software in the prerequisite introductory materials engineering laboratory; however, this software is relatively user friendly in creating materials properties correlation plots, and this can be taught to students in about 30 minutes.

2. The current hardness experiment relates to several previous years' NEW papers [20-24] on various aspects of hardness testing.

3. Use of the CES software has been the topic of several past NEW papers [24-27].

4. At the suggestion of a reviewer for those who do not have access to a Vickers microindentation hardness tester, a set of scaled, higher magnification images of the hardness profile impressions could be obtained off-campus and photocopied for distribution to the students. Impression diagonal measurements then can be obtained with a dial caliper or possibly a ruler; appropriate conversion factors with explanation would be provided allowing calculation of hardness numbers. A second issue raised was the obtainment of samples; this can be solved by creating one or more metallographically-prepared sections taken from an old automobile aluminum radiator obtained from a junk/salvage yard. However, care must be taken to completely drain any residual antifreeze and flush the radiator before commencing sectioning.

5. The test block used in this work is a metallic material having a highly polished top surface and parallel bottom surface. Such standard reference blocks are available from the manufacturer of the tester and have a certified hardness specified with an upper/lower variation. Obtaining a

hardness reading within the permitted range of values for the block verifies that the tester is working properly and that a correct impression diagonal measurement technique is being used. Once this has been established, testing samples with unknown hardnesses can proceed with confidence. However if a large number of samples are being tested, it is desirable to perform intermediate and/or concluding calibration checks.

6. Prior to students making hardness measurements, the sample must be carefully aligned using a 40X objective lens (400X total magnification) so that the brazed joints were positioned vertically, and a hardness profile could then be obtained perpendicular to the joints.

7. In examining and interpreting computer-generated plots of this type, it is important to note that a given property for some materials has been estimated (and denoted as such) and not actually measured.

8. As specified in the procedure, this plot was generated from tabulated KHN data appearing in Wilson (Instron) Desk Chart 60 for most non-ferrous metals [16]. It is thought a reasonable estimate for tensile strength can be obtained since hardness values for Knoop and Vickers indenters are expected to be fairly close. However, it should be noted that the resultant tensile strength values <u>are only approximations</u> also because the values all involve extrapolations, although the data in Figure 4 is linear with the trend line $R^2 = 0.99$.

9. Relating 500 g_f hardness data to 10 g_f data is not thought to be a significant issue in this case because only a modest indenter load effect was observed for a separate subsequent set of Vickers measurements in the center core regions for indenter loads ranging from 10 to 500 g_f (Figure 7).

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SOURCES OF SUPPLIES: The CES EduPack software is available from Granta Design Limited, 300 Rustat House, 62 Clifton Road, Cambridge, CB1 7EG, United Kingdom; phone (800-241-1546); FAX (216-274-9812); Web site (*www.grantadesign.com*).

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ABOUT THE AUTHORS:

Patricia B. Roy

Patricia is a senior materials engineering major at Loyola University Maryland. She has been recognized with the university's "First Year Engineering Achievement"award, and she received the prestigious Hauber Fellowship in Summer 2011 to perform research on a non-invasive technique to test the degree of sensitization of an aluminum alloy. Under the supervision of Dr. Robert B. Pond, Jr., she continues to engage in this and a second research project on crack propagation in an aluminum alloy. Patricia plans to submit a presentation for the annual Council on Undergraduate Research poster session held on Capitol Hill in Washington, D.C., where she will describe her aluminum sensitization work. With graduation on the horizon, she is considering options for her post-university years. Her focus is on graduate studies in materials engineering and world travel, and she hopes to combine these two interests.

Fiona M. O'Connell

Fiona graduated from Loyola in May 2012 with a major in materials engineering and a minor in general business. She is currently working as the assistant to the lab manager for "MT Group" which is a materials testing firm located in Farmingdale, New York (Long Island).

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Tom graduated from Loyola in May 2012 with a major in mechanical and materials engineering as well as a minor in mathematics. He is currently working for The DEI Group, a contracting engineering firm located in Millersville, Maryland, that provides solutions for improving reliability and maintenance strategies to manufacturing and processing companies with the primary focus on the energy sector. His main project is implementing a system to monitor vital equipment and improve future reliability on the new U.S. Navy fleet of combat ships.

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Eddie graduated from Loyola in May 2012 with a major in materials engineering and a minor in mathematics. He is currently working as the assistant to the vice president of operations for "MT Group" which is a materials testing firm located in Farmingdale, New York (Long Island). He conducts tests on building materials (e.g., asphalt, concrete, steel, and windows) and also does inspections on buildings and foundations.

Wayne L. Elban

Since 1985, Professor Elban has taught engineering courses at Loyola College (now Loyola University Maryland), including introductory materials science, materials science lab, mechanical properties of materials, transformations in solids, and engineering materials and manufacturing processes. He received a BChE with distinction ('69) and a PhD in Applied Sciences: Metallurgy ('77) from the University of Delaware and a MS in Engineering Materials ('72) from the University of Maryland, College Park. From 1969-1985, he was a research engineer at the Naval Surface Warfare Center, White Oak Laboratory, Silver Spring, Maryland. In 1992, he was a Fulbright scholar at the University of Strathclyde (Glasgow), Department of Pure and Applied Chemistry. From 2001-2003, he was a working visitor at the Smithsonian Center for Materials Research and Education, Silver Hill, Maryland. From 2008-2011, he was a guest worker at the National Institute of Standards and Technology, Gaithersburg, Maryland. He is a member of ASM International and the Society of Manufacturing Engineers.

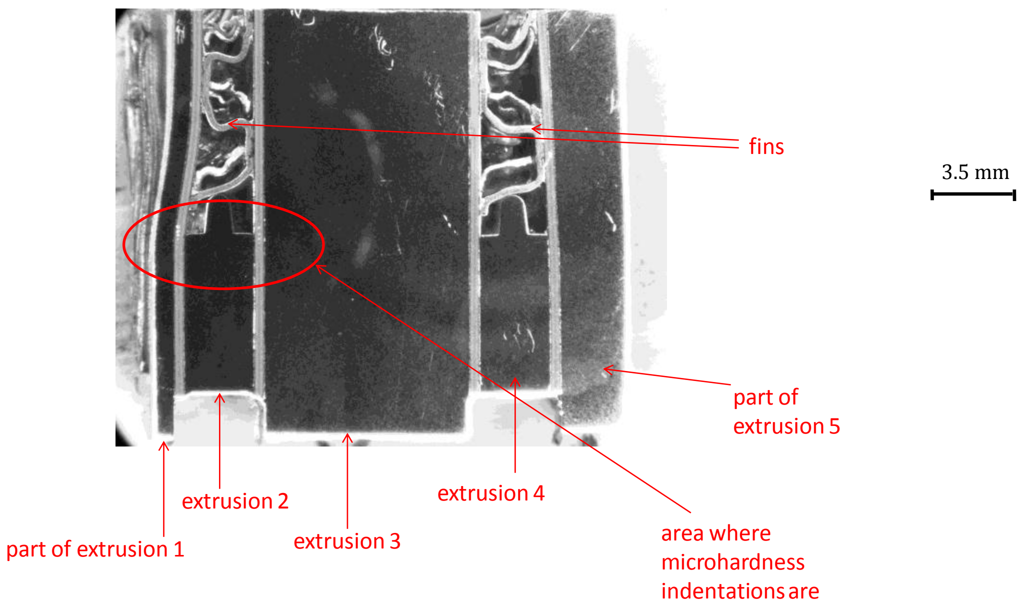

Figure 1. Low-magnification view of the cross-section of the manufactured aluminum alloy part (sample) showing various elements identified.

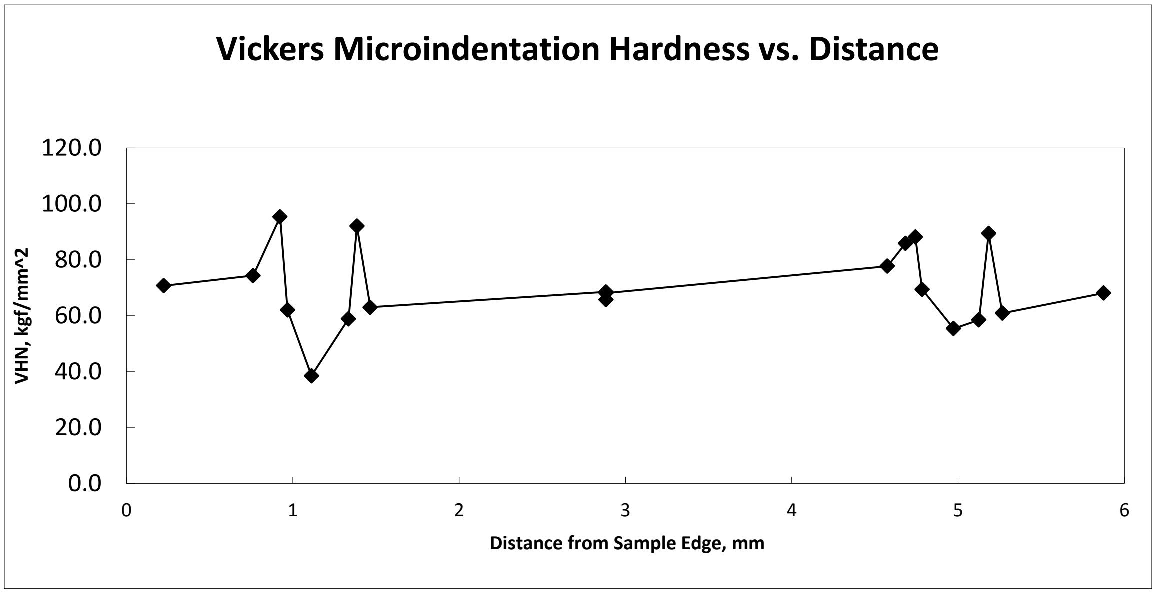

Figure 2. Vickers microindentation hardness profile of two brazed joint assemblies. Approximate locations of the four individual brazed joints are denoted with narrow paired red and blue vertical lines.

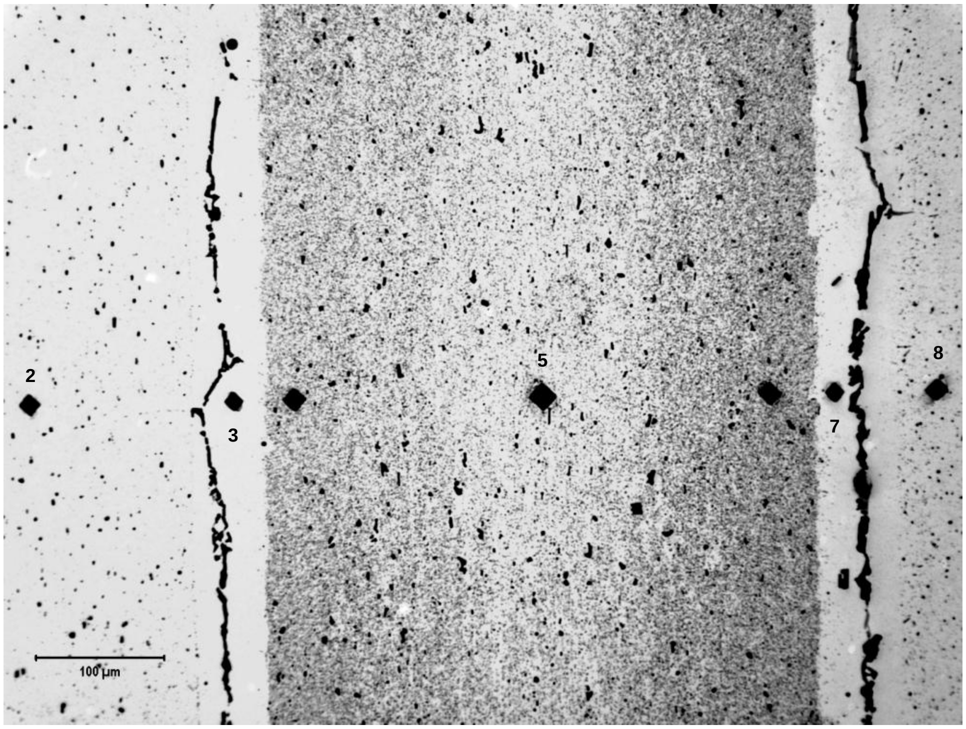

Figure 3. Photomicrograph of the left-hand brazed joint assembly showing the locations of Vickers microindentation hardness impressions #3 and 7 put in the brazed joints.

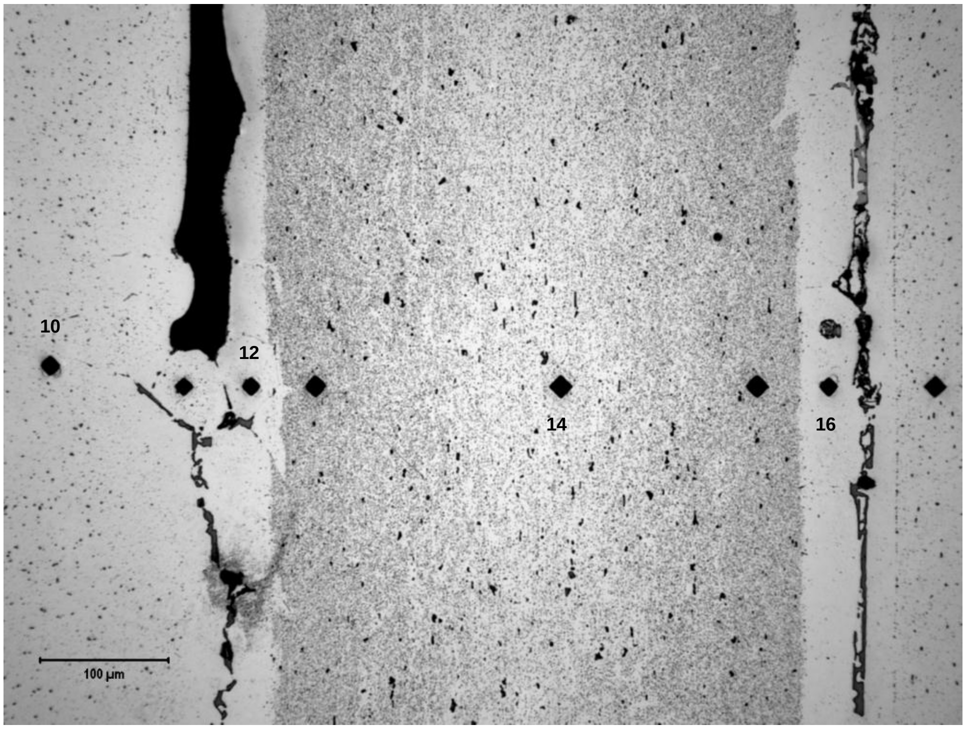

Figure 4. Photomicrograph of the right-hand brazed joint assembly showing the locations of Vickers microindentation hardness impressions #12 and 16 put in the brazed joints.

Table 1. Vickers microindentation hardness values with corresponding tensile strength estimates for impressions put in various locations in the brazed joint assembly sample.
[Measured standard test block hardness = 711.3 kg_f/mm^2 (average for two determinations) vs. manufacturer reported 710.5 ± 3.9 kg_f/mm^2]

Imp. #	VHN, kgf/mm^2	Tensile Strength Est., ksi	Location
1	70.7	29.3	1st extrusion
2	74.3	31.0	1st extrusion, near braze
3	95.3	40.7	**braze**
4	62.0	25.3	m. 3003 core sheet, very near braze
5	38.5	14.4	modified 3003 core sheet, center
6	58.9	23.8	m. 3003 core sheet, very near braze
7	92.0	39.2	**braze**
8	63.0	25.8	2nd extrusion, very near braze
9	68.5	28.3	2nd extrusion, center
19	65.7	27.0	2nd extrusion, center
20	68.1	28.1	2nd extrusion, center
10	77.7	32.5	2nd extrusion, very near braze
11	85.8	36.3	**braze**
12	88.2	37.4	**braze**
13	69.4	28.7	m. 3003 core sheet, very near braze
14	55.4	22.2	modified 3003 core sheet, center
15	58.5	23.7	m. 3003 core sheet, very near braze
16	89.4	38.0	**Braze**
17	60.9	24.8	3rd extrusion, very near braze
18	68.1	28.1	3rd extrusion, toward center

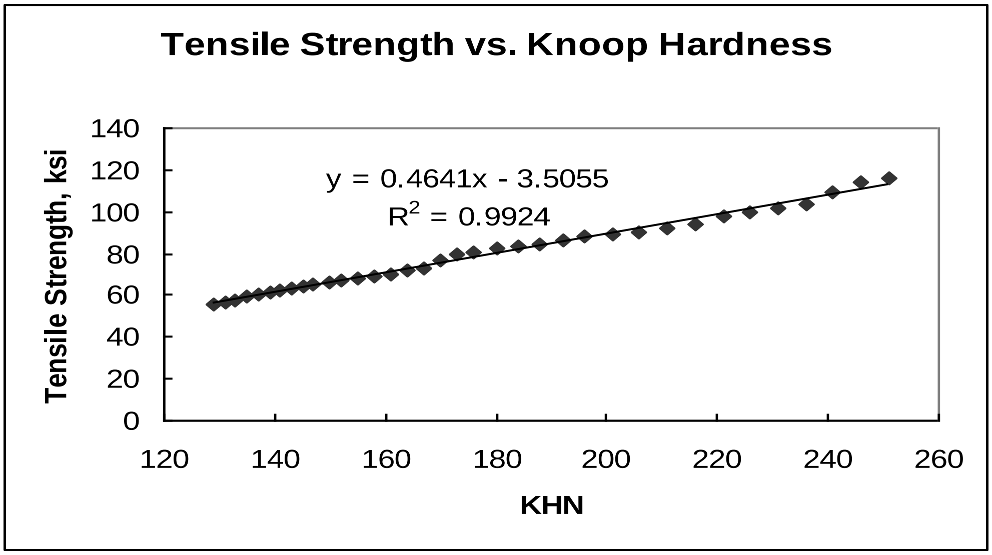

Figure 5. Tensile strength – Knoop hardness number (500 g_f) correlation obtained from Wilson (Instron) Desk Chart 60 (1992).

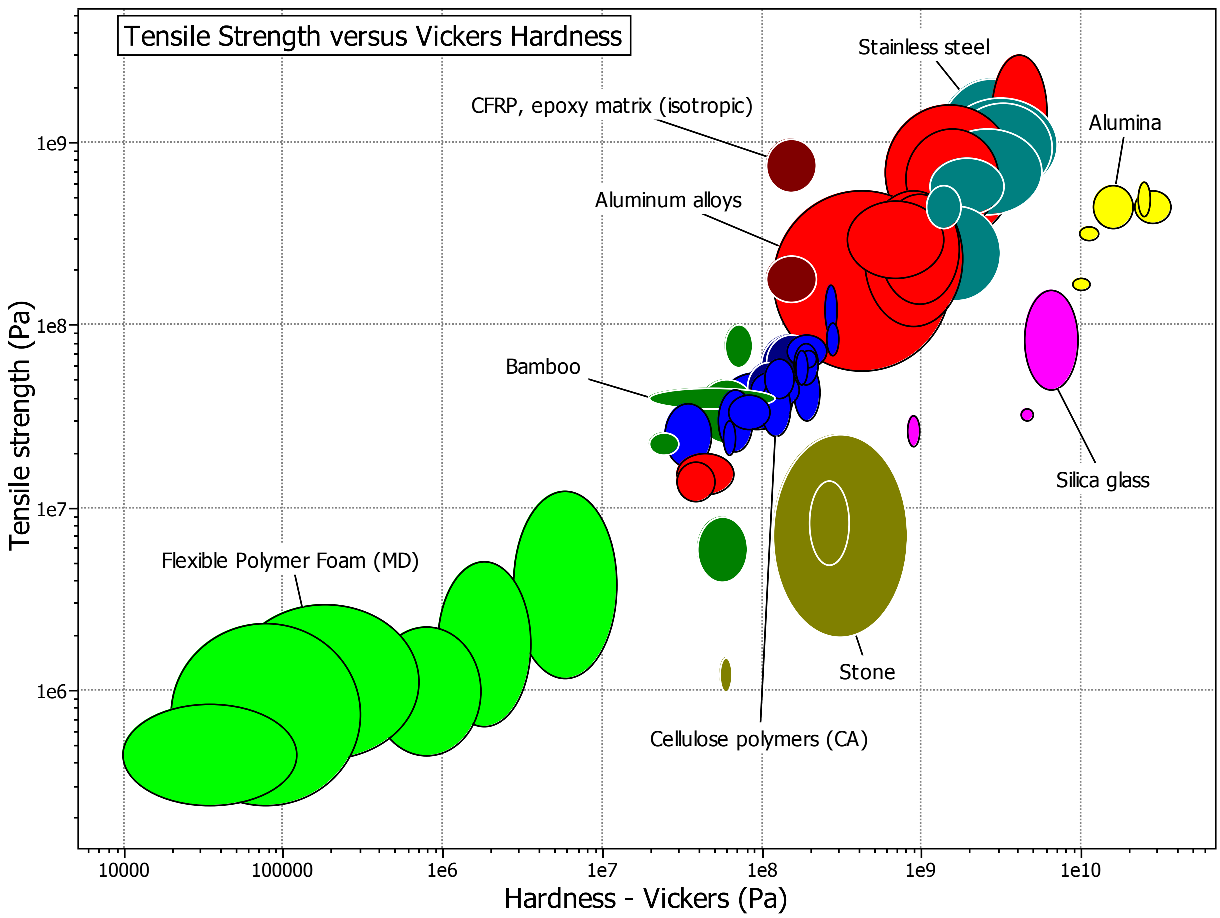

Figure 6. CES plot of tensile strength vs. Vickers hardness (obtained using Cambridge Engineering Selector EduPack 2012).

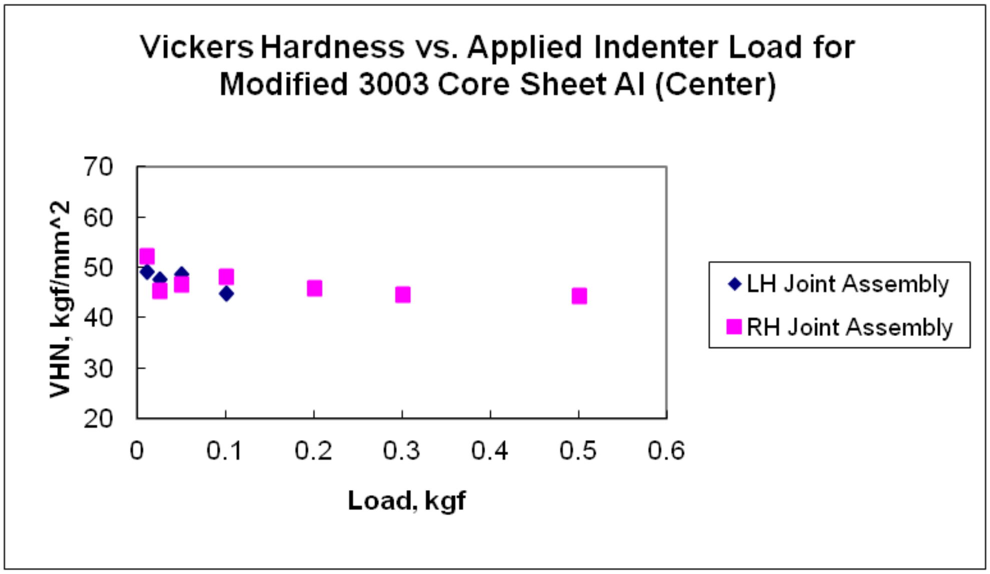

Figure 7. Indenter load effect for Vickers microindentations put in the center regions of the modified 3003 core sheet aluminum in the two brazed joint assemblies.

EVALUATION PACKET:

Student evaluation questions (discussion or quiz):

1. Describe how hardness pressure is calculated.
2. Explain why hardness testing can be used to characterize the mechanical state of material in and around phase boundaries (i.e., experimentally obtain a hardness profile for a set of braze joints).
3. Describe brazing and its advantages in joining metals/alloys.
4. Discuss what is accomplished by using a standard hardness test block.
5. Discuss two metallurgical reasons why the hardness in the braze joints in aluminum is elevated above the surrounding regions.
6. **(Optional)** Discuss the correlation that exists between tensile strength and hardness.

Instructor evaluation questions:

1. At what grade level was this module used?
2. Was the level and rigor of the module what you expected? If not, how can it be improved?
3. Did the lab work as presented? Did it add to student learning? Please note any problems or suggestions.
4. Was the background material provided sufficient for your background? Sufficient for your discussion with the students? Comments?
5. Did the lab generate interest among the students? Explain.
6. Please provide your input on how this module can be improved, including comments or suggestions concerning the approach, focus, and effectiveness of this activity in your context.

Course evaluation questions (for the students)

1. Was the lab write-up clear and understandable?
2. Was the instructor’s explanation comprehensive and thorough?
3. Was the instructor interested in your questions?
4. Was the instructor able to answer your questions?
5. Was the importance of materials testing made clear?
6. What was the most interesting thing that you learned?