AC 2012-4893: MECHANICAL CHARACTERIZATION OF SN AND SHAPE MEMORY ALLOY INTL NANOWIRES AS PART OF AN UNDERGRADUATE RESEARCH EXPERIENCE

Edwin Alexander Peraza Hernandez, Texas A&M University

Edwin Alexander Peraza Hernandez is an undergraduate student in aerospace engineering at Texas A&M University. He will receive his B.S. degree in Dec. 2012. He is currently an undergraduate researcher in the Shape Memory Alloy Research Team at Texas A&M University. His research interests include the fabrication, characterization, and modeling of micro and nano materials and structures. He is a member of AIAA, Tau Beta Pi, Phi Kappa Phi, Golden Key International Honour Society, Phi Eta Sigma, and Sigma Gamma Tau.

Dr. Kaushik Das, Texas A&M University, College Station

Kaushik Das was born in West Bengal, India in 1981. He received a M.Tech degree in aerospace engineering and a Ph.D. degree in engineering mechanics from the Indian Institute of Technology, Kanpur, India, and the Virginia Polytechnic Institute and State University, Blacksburg, USA, in 2005 and 2009, respectively. He is currently a postdoctoral Research Associate in the Department of Aerospace Engineering, Texas A&M University, College Station, Texas. His research interests include computational mechanics, smart structures and materials, nano-structured materials, and microelectromechanical systems.

Dr. Dimitris C. Lagoudas P.E., Texas A&M University

Dimitris C. Lagoudas received his B.S. from the Aristotle University of Thessaloniki, Greece in 1982. He then went to Lehigh University in Bethlehem, Penn., where he earned his Ph.D. in 1986. He pursued his postdoctoral studies from 1986-1988 at Cornell University and Max Planck Institute in Germany in theoretical and applied physics/mechanics. Next, he went to Rensselaer Polytechnic Institute in Troy, N.Y. and taught from Sep. 1988 to June 1993. Lagoudas arrived at Texas A&M University in July 1992, where he remains today. Lagoudas currently is the Department Head and the inaugural recipient of the John and Bea Slattery Chair in Aerospace Engineering at Texas A&M University. He serves as the Director for the Texas Institute for Intelligent Materials and Structures (TiiMS). His research involves the design, characterization, and constitutive modeling of multifunctional material systems at nano, micro, and macro levels using micromechanics methods developed to bridge the various length scales and functionalities including mechanical, thermal, and electrical. His research team is recognized internationally especially in the area of modeling and characterization of shape memory alloys. He has authored or co-authored about 365 scientific publications (150 in archival journals). For his scientific work on multifunctional materials, he received two best paper awards from ASME. He is co-author of a monograph on gauge theories of defects, and edited several special issues of journals and proceedings volumes in addition to a textbook on shape memory alloys co-authored with his graduate students. He has seven disclosures of invention and concepts developed for industry and a software license. During the past two decades he has published extensively on the subject of shape memory alloys with his students, postdoctoral associates, and colleagues and several of his journal papers are now considered classic papers in the field. The theoretical models that his research group developed have now been implemented and integrated into finite element analysis software, which have been used by many academic institutions around the world and also industry and government (Boeing, DoD, and NASA). He received the 2006 ASME Adaptive Structures and Material Systems Prize in recognition of his contributions to the modeling and characterization of shape memory alloys and their use in aerospace structures. Over the past two decades, his research has been supported by various government agencies including NSF, NASA, ONR, ARO, AFOSR, DARPA, DoE, and the state of Texas. He has collaborated with many industrial partners such as Bell Helicopter, Lockheed Martin, Northrop Grumman, Boeing, Schlumberger, and Tenaries. He has also worked with national labs, including DoD labs and NASA centers, either directly or through cooperative research and development agreements. He is an Associate Editor for the two main journals on smart structures and he helped organize numerous conferences through professional societies such as AIAA, ASME, SPIE, and SES, for which he served in various capacities. He is an alumnus of the prestigious Defense Science Study Group, and he has served on NRC panels. He also served as the Co-chair of NASA's Roadmap panel for nanotechnologies. He was the inaugural recipient of one of the two Ford Motor Company Professorships at Texas A&M, he is a TEES fellow, a TAMU Faculty Fellow, and he is an Associate Fellow of AIAA and a Fellow of ASME. He was selected as an SES Fellow in 2009. He served as an Associate Vice President for Research for Texas A&M University from 2001-2004, and as the First Chair of the Materials Science and Engineering Program at TAMU from 2001-2003.

Mechanical Characterization of Sn and Shape Memory Alloy InTl Nanowires as Part of an Undergraduate Research Experience

Abstract

This paper provides a description of an undergraduate student's summer project and an analysis of his overall learning and research experience, which took place during the summer of 2011. The first author, who was the undergraduate student, was supported by a summer research grant. One of the goals of this grant was to prepare students for graduate study and research. The student participated in an inclusive learning community of graduate students, postdoctoral associates, university faculty, and undergraduate researchers from the host university and from other universities. Student activities included preparation of research plans, weekly presentations to multidisciplinary research groups, preparation of progress reports and research papers, and research poster presentation. The student learned to operate state of the art laboratory equipment, such as scanning electron microscopes, energy dispersive spectroscopes, and nano-indenters, and computational software such as ABAQUS finite element analysis (FEA) simulation software. During the course of this project, a number of seminars focusing on research-based careers and graduate school opportunities were presented by leading faculties of the university.

The goal of the research work undertaken by the undergraduate student was to characterize the mechanical properties of Sn and shape memory alloy indium-thallium (InTl) nanowires embedded into cylindrical pores arranged along the thickness direction of anodic aluminum oxide (AAO) films. A broad range of nanotechnology applications requires one-dimensional nanostructures such as nanowires. Before any feasible application, the mechanical properties of such structures need to be characterized. The AAO films with constant pore diameter were fabricated using a two-step anodization. Molten Sn or InTl was injected into cylindrical pores of the AAO films to form the composites. Nanoindentation was performed on bulk Sn, bulk InTl, AAO films at different steps of the composite fabrication, and the finished composites. The force-displacement curves from the nanoindentation tests were analyzed to characterize the elastic properties of the specimens. The results showed that the indentation modulus of the AAO films decreased after heating and pressing of the films during the fabrication process. The reasons may be formation of nanocracks due to thermal and compressive stresses during heating and pressing of the films, respectively. The indentation modulus of the composites was lower than the bulk metals and the AAO films with empty pores. This may be due to nanocracks formed on the AAO film during the crystallization of the metal inside the pores.

Introduction

The project took place during the summer of 2011 and was funded by an undergraduate summer research grant. The purpose of the program was to involve high-achieving students who have completed their sophomore year of study in an intensive ten-week program involving research activities to motivate them to obtain higher education and research careers. Undergraduate research programs of other disciplines in the past have shared such objectives[1-3]. The first author of this paper, who was a junior undergraduate student during the program, was selected through a highly competitive selection process to work with a research group on multifunctional materials and Shape Memory Alloys (SMA) at the Aerospace Engineering Department at Texas A&M University. The selected student made a contribution to ongoing faculty research and gained appreciation for and an interest in research.

The student participated in a multidisciplinary learning community of graduate students, postdoctoral associates, and other undergraduate researchers. He was advised and mentored by a faculty member, and he had the opportunity to participate in an ongoing research project. This gave the student a flavor of graduate school and graduate study. The undergraduate student learned a number of research skills, such as operation of state of the art laboratory equipment: scanning electron microscopes (SEM), energy dispersive spectroscopes (EDS), and nanoindentors, and computational software, which included ABAQUS FEA simulation software. The student developed technical paper writing and presentation skills during different portions of the summer research program. Among the activities were the preparation of a research plan at the beginning of the summer, weekly presentations to faculty members and to a multidisciplinary group of student participants from different departments and various universities of US, preparation of progress reports, and a final research paper and research poster presentation at the end of the summer. The program also included a preparatory course for the standardized test for graduate school program applications (i.e. GRE) and several seminars on student-mentor interactions, graduate school opportunities, and research based careers.

Program Overview

The purpose of the undergraduate summer research grant program was to involve outstanding students who had completed their sophomore year and were interested in pursuing graduate studies. Eligible students should have interest and desire to actively participate in ongoing research projects, a minimum cumulative GPA of 3.25, and major in engineering or related science or math curriculum. Students were required to participate in all activities for the 10-week program, not being enrolled in other courses during the entire period other than the required independent study/research course, commit at least 40 hours a week to the program including sponsored activities and deliver the required assignments during the period. Students obtain a stipend, tuition and fees for one credit hour of a required independent study/research and housing allowance for their summer participation.

Learning Objectives

The primary focus of the program was to show the student how research in an academic environment works and to prepare the student for graduate school. Objectives were that the undergraduate student would experience working with a team of graduate students on a research program to learn research skills, learn to use state of the art experimental facilities, improve technical writing skills, and presentation skills. It is expected that the undergraduate student will make a significant contribution to the ongoing research on multifunctional materials and gain interest in the subject.

Technical background

Nanostructures such as nanoparticles, nanotubes, nanowires, and thin films have attracted much interest due to their potential applications in small scale devices and their enhanced properties compared to bulk materials[4-5]. For instance, nanowires have applications as gas sensors, resonators, mechanical dampers and light sensors[6-8]. Nanowires possess attractive mechanical and electrical properties[7-8]. Experimental studies have shown that Sn nanowires have unique superconductivity and magnetization properties which are not present in bulk Sn[8-9].

Various nanostructures with unique properties can be used to make multifunctional materials and nanoelectromechanical systems. A great amount of research has been performed on multifunctional materials such as piezoelectrics, magnetostrictives, thermoelectrics, electrostrictives and shape memory materials[10]. These materials are part of a family of materials called active materials. Active materials exhibit couplings between various forms of energy. For instance, shape memory alloys (SMAs) couple mechanical and thermal energy. From the family of active materials, SMAs have the highest actuation energy density defined as the product of actuation strain and actuation stress[11].

One distinctive property of SMAs is their reversible solid to solid phase transformation from a high temperature phase known as austenite to a low temperature phase known as martensite. Each phase has a different crystal structure and therefore different properties. Transformation can also occur by application of a sufficiently high stress to the material in the austenitic phase. The result of this transformation is a fully detwinned martensite phase created from the austenite phase. If the temperature is sufficiently high so the SMA is completely in the austenite phase at zero stress, a complete shape recovery is observed upon unloading to austenite. This SMA material behavior is called the pseudoelastic effect[12].

One drawback of the SMAs is the relatively low actuation frequency of bulk SMA structures (typically <1 Hz) compared with other active materials. This low actuation frequency is due to the generation of the latent heat of transformation. In order to allow fast phase transformation from austenite to martensite, SMAs need to have sufficiently high heat transfer rate to remove the latent heat of transformation. However, bulk SMA structures do not allow for a sufficiently high heat transfer rate for quick release of the latent heat. One possible solution to the problem of dissipation of heat is to reduce the dimensions of the SMA to the nanometer scale. It is expected that nanoscale SMAs will have a higher heat transfer rate due to the high surface area to volume ratio and therefore allow a much higher actuation frequency[11]. By allowing a higher actuation frequency, SMAs will potentially be able to substitute electrical and mechanical nano actuators and reduce the volume and complexity of the actuation devices.

In this research project, the elastic behavior of Sn nanowires and InTl SMA nanowires was studied by nanoindentation tests of such nanowires embedded in AAO films. The mechanical pressure injection method was used to fabricate the nanowires[13]. Molten Sn or InTl was injected into cylindrical pores, which were arranged along the thickness direction of the AAO films, to form the composites. Nanowires of different diameters were fabricated using AAO films with pores of different diameters. Nanoindentation was performed and analyzed on bulk Sn, bulk InTl, AAO films at different steps of the composite fabrication, and the finished composites. The studied specimens had approximately 15 nm and 40 nm average pore diameters (AAO15 and AAO40, respectively).

The following two sections present a brief description of various activities and the experimental and data analysis steps carried out by the undergraduate student. The subsequent section presents his assessment of the summer research program and its impact on his career plans after completion of undergraduate study.

Student activities

There were five main assignments due at different times of the summer program. The first assignment was preparation of a research plan. This assignment was due after the first week of the program. The student and his mentor worked together and prepared an outline of the student's research objectives and activities. The second assignment was preparation of a midterm progress report. On this assignment, the student provided a summary of the work done during the first half of the program and reported any difficulties or problems they faced. The third assignment was writing of an abstract of their work near the end of the program. The abstract summarized the work and results obtained. The fourth assignment was preparation of a final technical report at the end of the program. This assignment presented in details the complete work done by the student and the results obtained. It was expected that the report should have a very high quality and it should be publishable in a journal or a conference. The final assignment was a poster presentation. The student created a porter summarizing his research and presented it during a poster presentation session attended by undergraduate students, graduate students and faculty members of the school of engineering. In addition to these assignments, the student gave a short presentation of their progress every week to a group of undergraduate and graduate students and faculty members of the department. Such presentations served as preparations for his main poster presentation and helped improve his oral presentation skills. The student also attended a GRE preparation course and several seminars. The seminars' topics included graduate school applications, federal funding opportunities, fellowship application processes, national laboratories, and effective poster presentations.

Description of the technical aspects of the project

Nanowires and Composites Fabrication

Bulk Metal Fabrication:

The preliminary step before using mechanical pressure injection to create the nanowires was to fabricate the bulk materials. Sn granules were purchased (Alfa Aesar, 99.9% metal basis) and bulk InTl alloy with a composition of In-23.5at% Tl was fabricated. InTl alloys with 18at% Tl to 30at% Tl show SMA properties[14]. For this range of compositions the melting temperature is about 160°C. The phase transformation temperature of In-23.5at% Tl is about -9°C[15]. Since the nanoindentation experiments were performed at room temperature (~22°C), an In-23.5at% Tl alloy which was expected to be in a fully austenite phase at the room temperature was fabricated.

In order to fabricate the alloy, Indium ingots (Alfa Aesar, 99.999% metals basis) and Tl granules (Alfa Aesar, 99.999% metals basis) were mixed in appropriate weight percentages by measuring their masses using an electronic balance. The mixture of In and Tl was sealed in a vacuum quartz tube and melted in a muffle furnace at 400°C for 48 hours and then homogenized at 125°C for 72 hours, changing the position of the tube every 24 hours. The resulting In-23.5at% Tl ingot was removed from the quartz tube by carefully breaking the tube. The fabrication process provided the student with hands on alloy fabrication experience which is not provided during standard engineering or freshman chemistry courses.

Mechanical Pressure Injection:

The mechanical pressure injection method was used to fabricate the AAO-nanowire composites. Various metallic nanowires (Sn, Bi, In) have been fabricated previously using this method[16]. An outline of this process is presented in figure 1. In brief, a disc shaped metal was placed over the AAO sample inside a heating chamber. The chamber was then vacuumed and heated up to the melting temperature of the metal (~230°C for Sn, ~160°C for InTl). Then, this metal inside the heating chamber was pressed using piston and a conventional hydraulic jack. The chamber was subsequently cooled down to allow the molten metal inside the pores of AAO to crystallize. Then the residual metal was carefully removed from the top of the AAO film by peeling it off by hand. A detailed description of this method was presented by Chen et al[13].

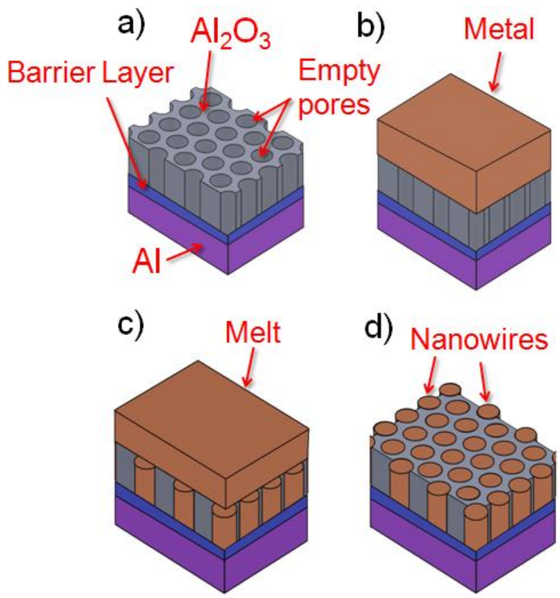

Figure 1. Schematic representation of the fabrication process of the composite: (a) porous anodic aluminum oxide (AAO) and the Al substrate attached to the AAO, (b) set-up before injection, (c) nanowires formed after melting and pressing of the metal, (d) nanowires inside the pores of AAO after the residual metal was carefully peeled off.

Fabrication of nanoporous AAO:

Anodic aluminum oxide films were fabricated by oxidation of aluminum films in two step anodization process[17]. A flat platinum (Pt) sheet and an aluminum (Al) film were used as the cathode and the anode, respectively, for the anodization. The size of the pores of the AAO can be controlled by manipulating the voltage and the electrolyte. AAO films with 15 nm and 40 nm average pore diameter were fabricated for the present study. To fabricate AAO15, the first anodization was performed for 1 hour at 18 V using 1.876 M H_2SO_4 as the electrolyte. The Al_2O_3 formed on the Al films during the first anodization was dissolved by submerging the films in a mixture of 0.4 M H_3PO_4 and 0.2 M H_2CrO_4 for 24 hours. By dissolving the layer of Al_2O_3, a flat surface for the AAO films was achieved. The second anodization was performed for two hours with the same voltage and electrolytes as the first anodization. The resulting depth of the pores was about 10 μm. For the AAO40, the same process was followed with the anodization voltage of 40 V and an electrolyte of 0.3 M $H_2C_2O_4$ (Oxalic acid).The fabrication of porous AAO provided the undergraduate student the experience of manufacturing nanomaterials with chemical-electrical-mechanical coupled processes.

Characterization

Nanoindentation experiment:

Nanoindentations[18] on the bulk metals, AAO films at different steps in the fabrication and the AAO-nanowires composites were performed using a nanoindenter with a diamond conical tip with a spherical shaped edge of 10 μm radius. Nine different maximum loads between 6000 μN and 8000 μN were applied at different locations to the bulk metals, the AAO films at different steps of the fabrication, and the finished composites.

The student was trained in the usage of SEM, EDS and nanoindenter. Then the experimentation was performed. The training and experience in such devices complements the theoretical courses of materials science.

Results and discussion

Characterization of the pore size and porosity of AAO films:

Figure 2 shows the SEM images of the AAO15 and AAO40 films. The average pores diameters were found to be approximately 15 nm and 40 nm. The porosities are presented in table 1.

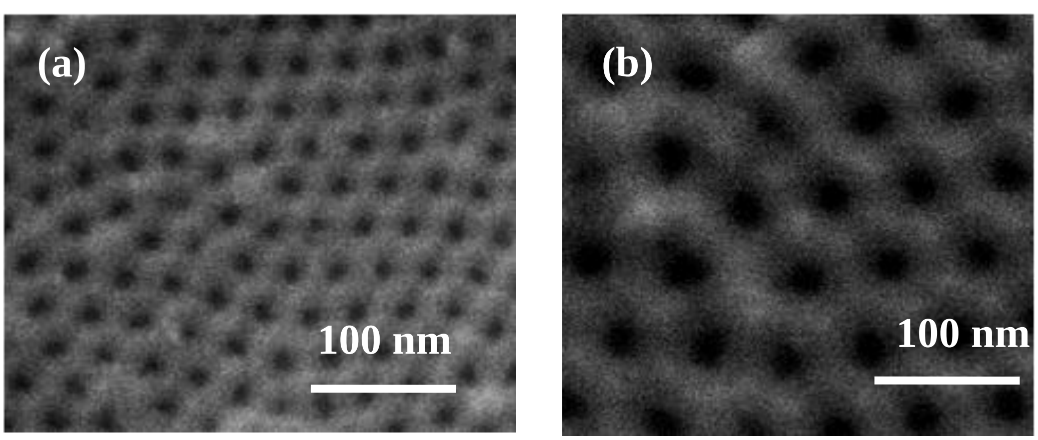

Figure 2. SEM images of AAO structures with sample: (a) AAO15, (b) AAO40.

Table 1 Porosity

Specimen	Diameter [nm]	Porosity from image processing
AAO15	15	10 %
AAO40	40	20 %

Nanoindentation of the AAO films at every step of the fabrication and the AAO-nanowire composites:

Figure 3 shows the SEM images of the AAO15-Sn and AAO40-Sn composites. From these images, it is observed that the majority of the pores were filled with metal during the mechanical pressure injection. A filling ratio of 90% was estimated for both specimens.

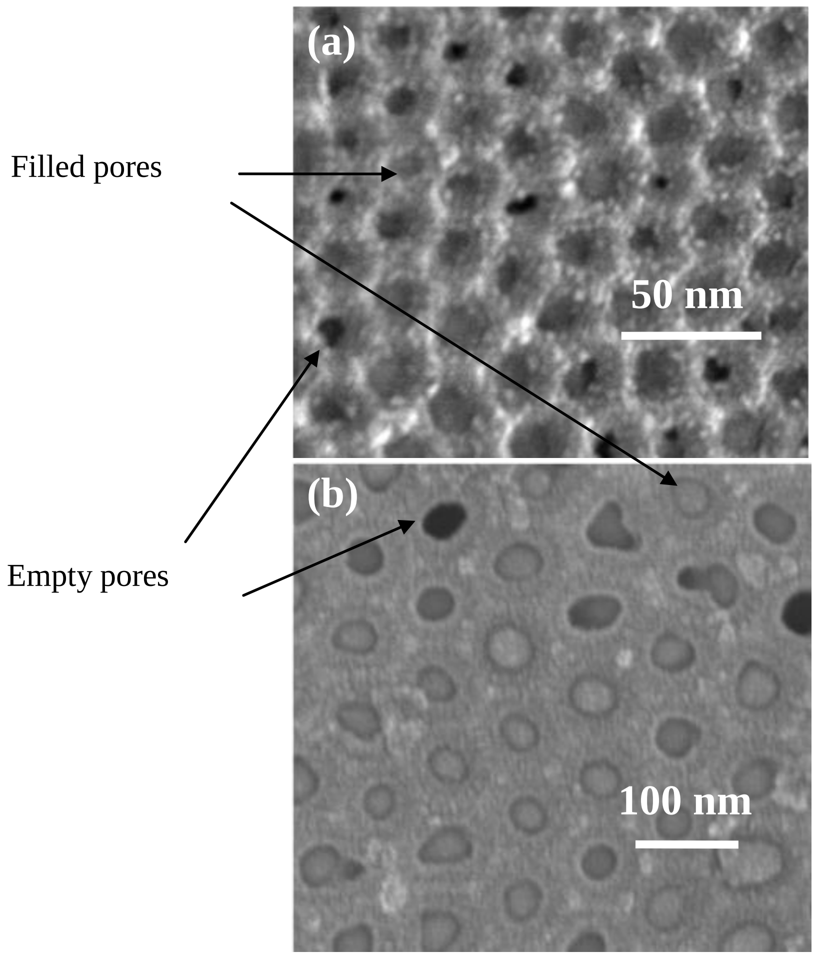

Figure 3. SEM images of AAO-Sn composites: (a) AAO15-Sn, (b) AAO40-Sn.

Figure 4 shows the nanoindentation curves for AAO15 at different steps of the fabrication. A decrease in the unloading slope was observed after heating the AAO films and further decrease was observed after pressing the films. The nanoindentation results of the AAO15 films and the composites based on AAO15 are presented in figure 5. A trend of decrease of indentation modulus (E^*) in every step of the composite fabrication was observed on the presented range of maximum loads.

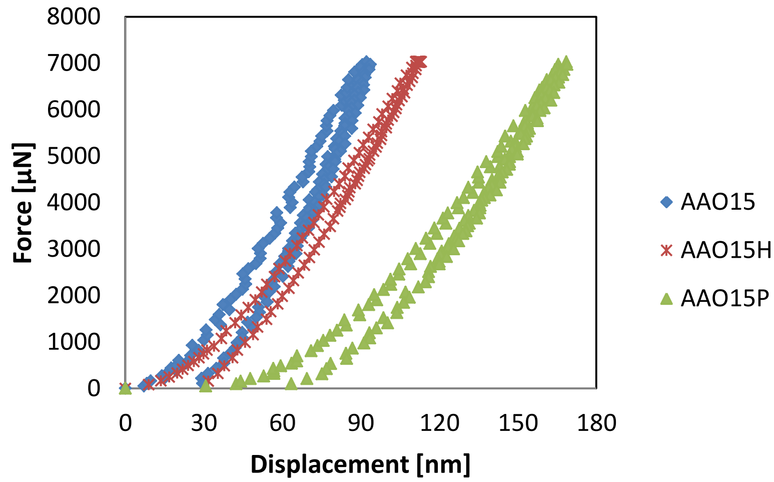

Figure 4. Nanoindentation curves of AAO15 at different steps of the mechanical pressure injection: AAO15, film after anodizations; AAO15H, film after heated up to ~230°C; AAO15P, film after being pressed at same pressure as required for infiltration.

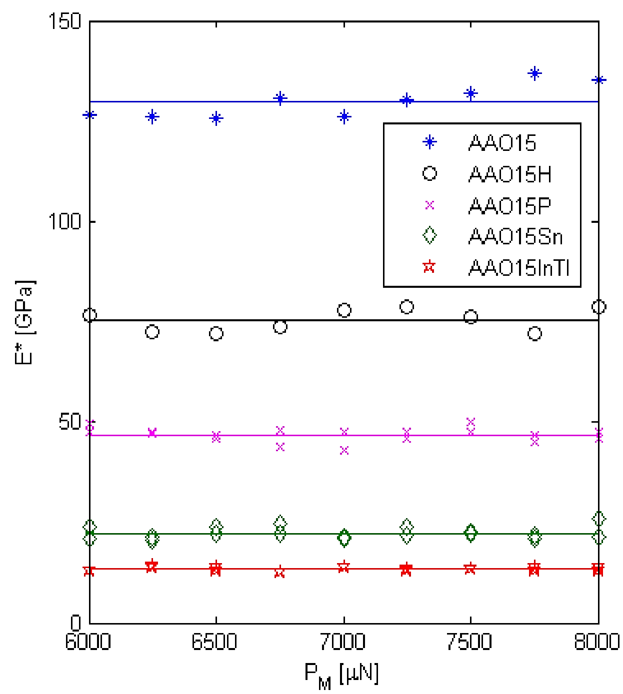

Figure 5. E^* vs. maximum indentation load (P_M) for AAO15 during the steps of fabrication: AAO15, film after anodizations; AAO15H, film after heated up to ~230°C; AAO15P, film after being pressed at same pressure as required for infiltration; AAO15Sn, finished composite with embedded Sn nanowires; AAO15InTl, finished composite with embedded InTl nanowires.

For all the AAO films at different steps of the fabrication and both composites, E^* was lower for the films with 40 nm pore diameter than the 15 nm diameter. This was due to the higher porosity on the AAO40 films. During all steps the decrease of E^* was greater on the AAO15 film than the AAO40. The temperature applied to the AAO films during the mechanical pressure injection was ~230°C and was applied for a short time (<40 min). Therefore, the decrease on E^* after being heated was due to possible nanocracks formed by thermal stresses on the sample due to the amorphous nature of the AAO films. The decrease of E^* due to heating on the AAO15 was about 41% greater than the one of AAO40.

In order to estimate the effects that the pressing had on E^* of the film alone, the mechanical pressure injection was implemented for the AAO films without pressing metal inside. The heated and pressed AAO films were then examined using indentation. The reduction on E^* due to pressing was about 33% greater on AAO15 than AAO40, due to the higher pressure applied to the AAO15 to reach an acceptable filling ratio. A reduction of E^* was also appreciable after the composites were finished. This was due to nanocracks created by stress on the pore walls during the crystallization of the metal inside the pores.

Student Assessment

The first author felt that the overall experience of the summer research was very enjoyable. He learned technical writing, presentation, and laboratory skills, which are not taught thoroughly in the standard curriculum. The interactions with a faculty mentor, graduate students and other undergraduate students from the host university and from other universities from all over US, who participated in the summer research program, were very fruitful. The seminars and research meetings gave him a flavor of a graduate study by being immersed in the research group for the ten-week period. The experience provided a positive image of graduate schools and encouraged him to enter a graduate program after obtaining his bachelor’s degree. The interactions with graduate students and postdoctoral associates during the program were very productive. The graduate students and postdoctoral associates gave him feedback and criticism on the technical presentation and writing. These criticisms greatly helped him to improve the quality of the presentations and writings.

The student acquired several research skills during the program. Among them are writing and presentation skills, development of a research plan, progress report writing, research paper writing, and preparation of research posters. The graduate student and postdoctoral mentors helped him learn the skill of writing and/or explaining difficult research topics, avoiding unnecessary details, and providing proper references in paper writing and/or presentations. He also for the first time had the opportunity to learn and use state of the art experimental facilities for nano-scale material fabrication and characterization tools, such as fabrication of alumina template with nanopores, template assisted nanowire fabrication, scanning electron microscopes, nano-indenters, and energy dispersive spectroscopy. Learning and using these equipment, which are generally only accessible to graduate students, was exciting and a very unique opportunity, which would not have been possible during the standard semester activities.

The weekly research presentations with the department faculty and other undergraduate research assistants provided the student with a comprehensive discussion of diverse research projects in the aerospace engineering discipline. The topics of the other participants' projects covered areas such as aerodynamics and propulsion, dynamics and control, and materials and structures.

Overall, the summer research experience motivated the undergraduate student to pursue a PhD degree. He feels that the positive experience of the summer research program and the various seminars given by eminent professors greatly motivated him towards a research career. The summer program also included preparatory course for the GRE. The student learned various effective techniques to improve his performance on the GRE. Among different topics discussed during the course, such as learning techniques to improve vocabulary and analytical skills, he found the techniques for stress management before and during the test to be particularly useful. This preparatory course for GRE helped him train for the exam, which is a prerequisite for successful admission to a graduate program.

Conclusions

This research activity was part of a summer undergraduate research grant. The first author, who is an undergraduate student, learned and used state of the art experimental equipment and acquired valuable research skills, such as writing technical reports and papers, preparation of research plans, and presentation of research findings to research groups. Overall, this summer research experience motivated the undergraduate student to pursue a PhD degree after completion of his undergraduate study.

During the project, the mechanical properties of AAO-nanowire composites were studied using nanoindentation. The effects that the fabrication process and porosity have on the mechanical properties of the composite were studied. The results show that heating and pressing of the AAO films during the fabrication of the composites decrease their stiffness. This is due to formation of nanocracks caused by thermal stresses when heating and compressive stresses when pressing. Further decrease in stiffness was appreciated after filling the pores of the AAO films with melted metal to form nanowires during the mechanical pressure injection. The reasons may be stresses on the pore walls during crystallization of the metal that led to formation of nanocracks in the AAO film. The nanoindentation curves of AAO-Sn and AAO-InTl do not show any major difference. The reason may be the low volume fraction of metal in the composites.

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