

# A Novel Hybrid Heating Method for Mechanical Testing of Miniature Specimens at Elevated Temperature

**Lin Li**

Department of Mechanical and Aerospace Engineering,  
North Carolina State University,  
Raleigh, NC 27695-7910  
e-mail: lli24@ncsu.edu

**Gracious Ngaile**

Mem. ASME

Department of Mechanical and Aerospace Engineering,  
North Carolina State University,  
Raleigh, NC 27695-7910  
e-mail: gngaile@ncsue.du

**Tasnim Hassan**

Department of Civil, Construction, and  
Environmental Engineering,  
North Carolina State University,  
Raleigh, NC 27695-7908  
e-mail: thassan@ncsu.edu

*A novel hybrid heating method which combines the conventional electric-resistance specimen heating with microcoil heating of specimen ends to achieve uniform heating over the gauge length is presented. Resistive heating of a miniature specimen develops a parabolic temperature profile with lowest temperature at the grip ends because of the heat loss to the gripper. Coil heating at the specimen ends compensates for this heat loss resulting in uniform temperature distribution over the central segment of the specimen. Thermo-electric finite element simulations were carried out to analyze the transient and steady temperature distribution in miniature specimens followed by experimental validation.*

[DOI: 10.1115/1.4035954]

## Introduction

In recent years, mechanical testing of miniature specimens has drawn significant attention [1–7]. The increasing need of miniaturization of mechanical and electronic components in many industries, such as, biomedical, cell phone, and micro-electro-mechanical systems, has increased the demand for miniature specimen testing [8–11]. The development of microfabrication techniques for producing microparts requires the knowledge of material properties which can only be determined through miniature specimen testing. Another factor for increased demand of miniature specimen testing is the determination of the remaining life of critical components working under extreme loading and environmental conditions [4,6,12]. Miniature specimen allows determination of material properties from a small amount of material normally produced while designing and developing new alloys. When mechanical testing of materials from a component in service needed to be performed usually small volume of materials can be extracted, and hence miniature specimen testing is imperative [13].

To date, there are numerous testing methods and systems that have been developed for mechanical testing of miniature

specimens [1–7]. Most of these systems, however, can perform mechanical testing at room temperature. There are many applications that call for accurate determination of material properties at elevated temperatures. For example, microforming at elevated temperature enhances homogeneous deformation; hence, determination of material properties at elevated temperature is essential for the development of simulation models [14]. In fusion welding, material properties at elevated temperature are needed to simulate residual stresses and structural performance of welded components. Furthermore, remaining life estimation of structures, welding joints, and components subjected to thermal loading can be performed by extracting small volume of materials for miniature specimen testing. Finally, in situ microstructure investigations within scanning electron microscope (SEM) while performing mechanical testing provide additional information for multiscale modeling and condition assessment [15–19].

Heating methods available for miniature specimens testing of metals and alloys are resistive heating and chamber heating [20–22]. The widely used induction heating systems are not available for miniature specimen testing. As demonstrated below, a primary set back of the resistive heating of miniature specimen is to achieve uniform temperature over the gauge length. In order to have relatively uniform temperature distribution over the specimen central segment, specimen lengths need to be increased, which defeats the purpose of miniature specimen testing. Uniform temperature distribution of miniature specimens can be obtained through chamber heating, but this method may not be feasible if the material test setup is to be integrated in a SEM system for in situ studies as the specimens need to be accessible to the SEM detectors and electron guns. Induction heating system can be developed for uniform heating of miniature specimens, but the electromagnetic fields from this method may interfere with the electron beams from the SEM resulting in image distortions. Hence, among all the miniature specimen heating techniques available, the resistive heating seemed best suitable for in situ SEM testing using a miniature test systems. However, as stated above and demonstrated below, this method is incapable of uniformly heating miniature specimens. Hence, this study developed a novel hybrid heating method combining resistive heating and specimen end microcoil heating. Analytical development and experimental validation of the hybrid heating method is presented below to demonstrate the potential of this novel heating technique.

## Resistive and Hybrid Heating Methods

A miniature test system is under development to perform high temperature, multiaxial, monotonic, and cyclic loading tests of miniature tubular specimens with 1 mm outer diameter and 0.69 mm inner diameter. The smallest gauge length of the miniature specimens will be in the order of 3 mm. In the testing process, the specimen ends are clamped by the grippers (see Figs. 1(a) and 1(b)) through which mechanical loads and current are applied. Electric-resistance specimen heating is obtained by conducting electric current through the specimen to generate Joule heat. Since the specimen is small and the heating power is low, the grippers can be cooled by natural convection when performing material testing at low temperatures. At higher temperatures, grippers can be cooled by running water through coolant grooves machined in the grippers (see Fig. 1(b)). It should be noted that the grippers must be cooled down to ensure that excessive heat is not transferred to the load cell. The coil heating system is composed of coils and ceramic insulators to heat the specimen ends as shown in Fig. 1(b) (exploded view of the specimen). As current flows through the coil, the Joule heat generated is conducted to the specimen via a ceramic spacer, which also acts as an electric insulator. This coil heating method by itself will not achieve uniform heating at the gauge length similar to the resistance heating method. However, when these two heating methods are combined uniform temperature heating of miniature specimens can be performed as needed for elevated temperature mechanical testing of materials.

Contributed by the Manufacturing Engineering Division of ASME for publication in the JOURNAL OF MICRO- AND NANO-MANUFACTURING. Manuscript received September 16, 2016; final manuscript received February 3, 2017; published online March 23, 2017. Editor: Jian Cao.

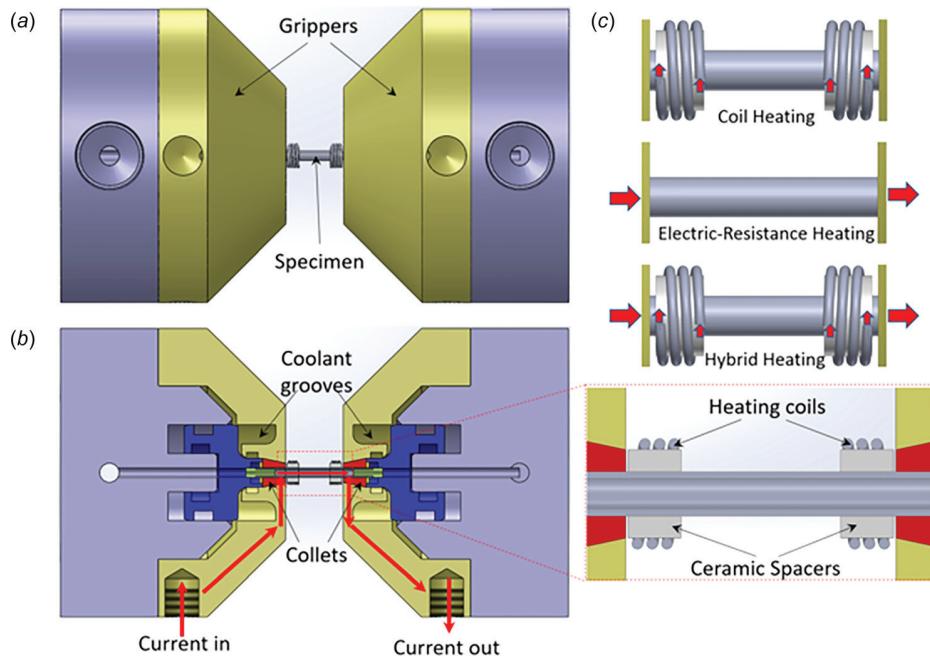


Fig. 1 The resistive, coil, and hybrid heating methods, (a) and (b) the grippers and specimen, (c) current input for coil heating, resistive heating, and hybrid heating

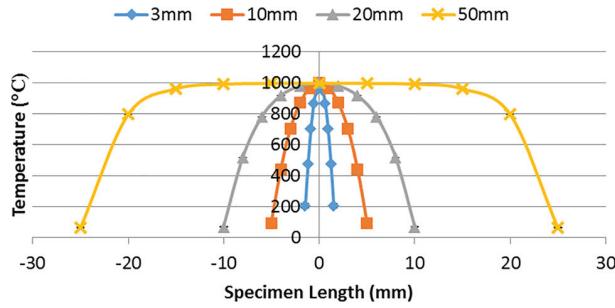


Fig. 2 Electric-resistance heating temperature distributions for different specimen lengths

The performance of these heating techniques is first simulated through finite element electrical-thermal analyses to demonstrate why uniform temperature can be achieved by the proposed hybrid heating method.

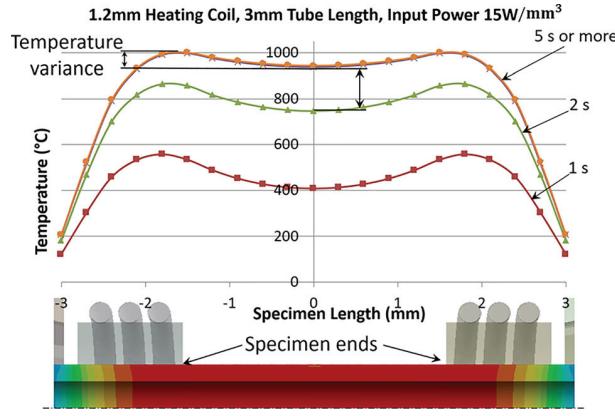


Fig. 3 Transient and steady temperature distributions of a 6-mm specimen by coil heating

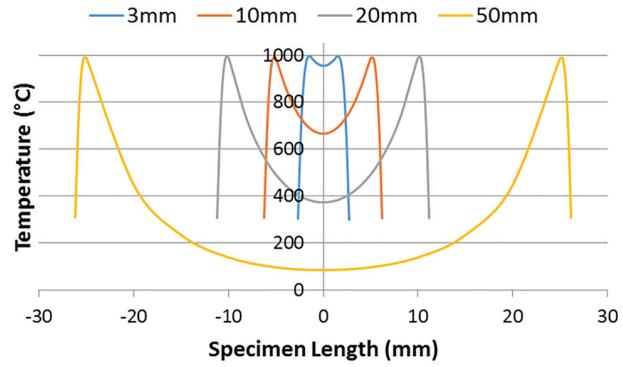


Fig. 4 The steady temperature distribution by coil heating for different specimen lengths

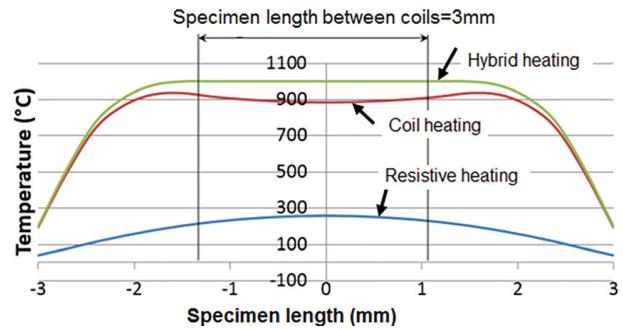


Fig. 5 The temperature distributions in a 6-mm-long specimen by three different heating methods

#### Finite Element Electrical-Thermal Analysis of Resistance and Coil Heating

In order to determine the temperature distributions by the resistance, coil, and hybrid heating methods, electrical-thermal finite element analyses were conducted. The analyses included heat loss

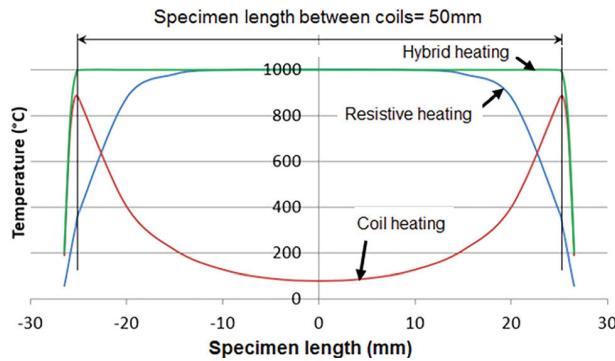


Fig. 6 The temperature distributions in a 50-mm-long specimen by three different heating methods

through radiation and natural convection. Variables such as, heating coil length, specimen length, power inputs, and the heating time to steady-state temperatures were investigated to better understand the influences of these variables on the heating methods. Electrical-thermal finite element analyses were conducted using the ANSYS WORKBENCH. Several assumptions were made to simplify the simulation process: (a) the connections between contact surfaces are set to bonded conditions with a thermal conductance  $0.1 \text{ W/mm}^2 \text{ K}$  [23], (b) the coolant groove surfaces are set to a constant temperature of  $22^\circ\text{C}$  to simulate water cooling, implying that water is pumped through the coolant grooves to maintain this temperature, and (c) thermal conductivity was taken as constant by averaging thermal conductivity values over a temperature range of interest. For example, below  $400^\circ\text{C}$  a thermal conductivity of  $15.1 \text{ W/m K}$  was used [24]. Stainless steel material was used for the grippers and specimen, whereas A2 tool steel was used for collets (Fig. 1). The material of the heating coils was Nichrome 80 and that of the ceramic insulators was Alumina 960.

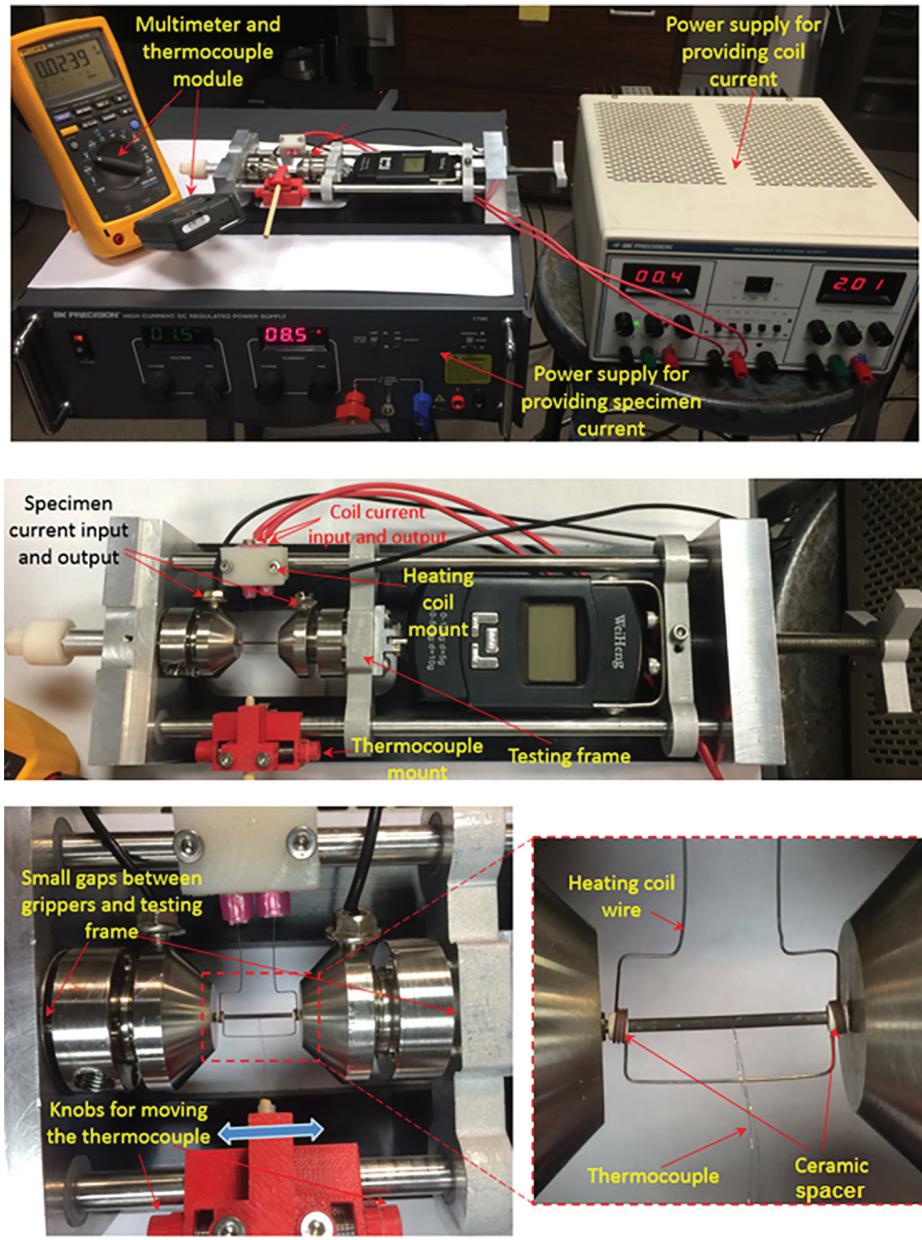


Fig. 7 The experiment setup for validation of the novel hybrid heating technique

**Finite Element Analysis of Electric-Resistance Heating.** In the thermal analysis of electric resistance specimen heating, the specimen was heated to a maximum temperature of 1000°C. The steady-state temperature distributions on the specimens are shown in Fig. 2 for different specimen lengths. As can be seen in the figure, the temperature distributions for shorter specimens are parabolic-shape with maximum at the specimen center, whereas that for longer specimens the temperature distribution at the central segment become uniform. The parabolic shape exhibited by shorter specimen is because the grippers act as heat sink. The simulation results (Fig. 2) show that the temperature profile for shorter specimens (6–10 mm) has nonuniform temperature distribution and hence the resistance heating method by itself cannot be used for elevated temperature mechanical testing of miniature specimens.

**Finite Element Analysis of Specimen End Coil Heating.** Transient temperature distributions simulated by the electrical-thermal analysis for specimen end coil heating (Fig. 1(c)) for a 6-mm-long tubular specimen are shown in Fig. 3. This figure shows temperature distributions at first, second, fifth, and 50th seconds. To heat the specimen to a maximum 1000°C, it took 15 W/mm<sup>3</sup> internal heat generation on the coils. The transient simulation results also show that a steady-state condition was reached by 5 s. The temperature variance between the maximum temperature and the temperature at the specimen center is 60°C for the 6-mm specimen; hence, coil heating method cannot be used by itself for heating miniature specimens.

The power input to the coils for heating the specimens to desired maximum temperature depends on the specimen length as shown in Fig. 4. When the specimen length is less than 10 mm, the required power input increased significantly with specimen length. But when the specimen length is above 10 mm, the power input will barely increase with the specimen length. It is also observed that the temperature at the center of the specimen decreases with the specimen length (Fig. 4). As shown in the figure, if the specimen length is short, the temperature distribution on the specimen center will have parabolic profiles with the minimum temperature located at the center of the specimen.

**Finite Element Analysis of Hybrid Heating.** The finite element analysis results presented above demonstrated that either the resistance or coil heating method is unable to heat miniature specimens with a uniform temperature distribution over the gauge length essential for elevated temperature mechanical testing. The

resistive heating method generates maximum temperature at the center of the specimen, whereas specimen end coil heating results in higher temperature around the ends of the specimen. Interestingly, when these two heating methods are combined (hence, called hybrid heating method) uniform temperature distribution can be achieved over the central segment of the specimens with different lengths. Figures 5 and 6 show the steady-state temperature distributions for two specimen lengths (6 and 50 mm) heated by the resistance, coil, and hybrid heating methods. In these figures, it is observed that for both the specimen lengths uniform temperature distribution is achieved at the central segment of the specimens using the hybrid method. The results shown in Figs. 5 and 6 were carried out for input power of 13.2 W/mm<sup>3</sup> for the coil heating system and 0.823 W/mm<sup>3</sup> for the resistance heating system, if used individually to achieve a maximum temperature of 1000°C.

## Experimental Verifications

The heating experimental setup is shown in Fig. 7. Two separate power supplies were used for coil and resistance heating of the specimen. A K-type thermocouple with a 0.05 mm wire gauge was used for temperature measurements. The thermocouple was connected to an 80 TK thermocouple module which was plugged on a FLUKE 189 multimeter to record the temperatures. The grippers were thermally isolated from the cross bars by using Alumina 960 ceramic insulator disks (6.5 mm diameter and 1.75 mm thickness). A specially designed thermocouple mount was used to precisely set and move the thermocouple along the specimen length.

Figure 8(a) shows measured temperatures generated by resistance heating for a 10-mm-long specimen and a current input of 12 A. As shown in this figure, the measured temperature distribution is parabolic along the specimen length with a maximum temperature at the specimen center. Similar temperature distribution is also observed in the finite element analysis results as shown in Fig. 8(a) but with a little under prediction. Figure 8(b) shows measured temperatures along with the finite element analysis results generated by specimen end coil heating for the same specimen with a current of 2.25 A. Again, both the measured and simulated temperature distributions are parabolic with a minimum temperature at the specimen center, and little over prediction by the simulation. Figure 9 shows the measured temperatures and corresponding simulations when the specimen was heated by the novel hybrid method. One experiment was carried out to achieve a maximum temperature of 500°C, using current inputs of 2 A and 8.5 A for coil heating and resistive heating, respectively. A second set of experiment was carried out to achieve a uniform temperature of 800°C, where current inputs of 2.25 A and 13.5 A were applied for coil heating and resistive heating, respectively. The measured temperatures generated by the hybrid heating clearly shows that uniform temperature can be achieved over the specimen gauge length (Fig. 9). The simulation results for the hybrid heating method, however, show slight parabolic shape.

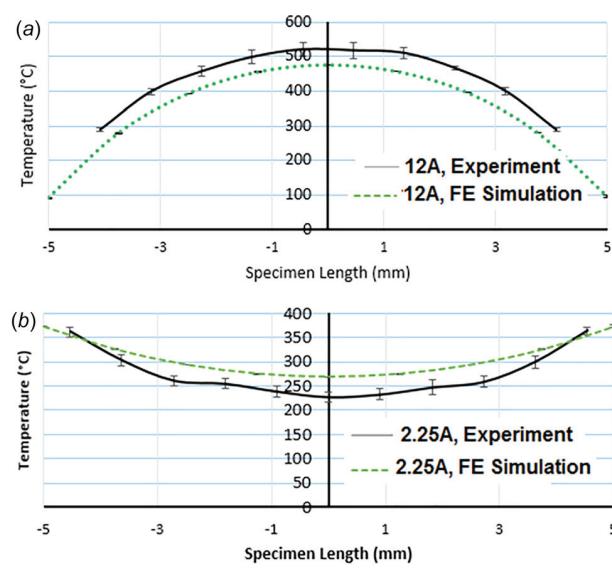


Fig. 8 Temperature distribution for a 10-mm-long specimen by (a) resistance heating and (b) specimen end coil heating

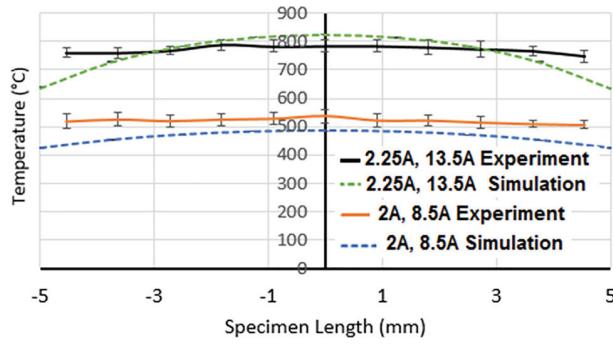


Fig. 9 Temperature distribution for a 10-mm-long specimen by hybrid heating

While the proposed hybrid heating method results in uniform temperate at the specimens gauge length, implementation of this technique in a miniature specimen testing will require a robust control module. For instance, tensile test results with an appreciable change in a cross-sectional area, which implies that the current density for resistive heating required to maintain the desired temperature will have to be changed. This will only be feasible if a feedback loop is incorporated in the system such that the current/input power is adjusted in real-time. Such a system is currently under development.

## Conclusions

Of the available heating systems, induction, electric-resistance, and furnace, only the resistance heating system can be implemented for the in situ elevated temperature mechanical testing of miniature specimens within SEM. However, the resistance heating method is unable to generate a uniform temperature distribution over the gauge length of miniature specimens. Hence, a novel hybrid heating method, which combines the electric-resistance and specimen end coil heating, is developed. In this hybrid method, two microheating coils are used to heat the specimen ends while heating the specimen through resistance heating to achieve a uniform temperature distribution over the gauge length of the specimen. For initial development of the hybrid heating method, electrical-thermal finite element analyses were conducted for resistance, coil, and hybrid heating systems. Based on the analysis results, an experimental setup was developed and performance of the novel hybrid heating system was validated to achieve the desired uniform temperature distribution in miniature specimens. The finite element analyses conducted and experimental validation revealed that the specimen length does not influence the temperature distribution by the hybrid heating system as was observed in case of the resistance and coil heating systems. Since the cross-sectional areas of the sample will change during mechanical testing, the current density for resistive heating required to maintain the desired temperature will also change. Thus, implementation of this heating technique requires a feedback loop, which is currently under development, to adjust the input current in real-time.

## Acknowledgment

The authors acknowledge the National Science Foundation funding for this Major Research Instrumentation Project (CMMI-1337952). Any opinions, findings, and conclusions or recommendations expressed in this article are those of the authors and do not necessarily reflect the views of the National Science Foundation.

## References

- [1] Lord, J. D., Roebuck, B., Morell, R., and Lube, T., 2010, "Aspects of Strain and Strength Measurement in Miniaturized Testing for Engineering Metals and Ceramics," *Mater. Sci. Technol.*, **26**(2), pp. 127–148.
- [2] Jaya, B. N., and Alam, M. Z., 2013, "Small-Scale Mechanical Testing of Materials," *Curr. Sci.*, **105**(8), pp. 1073–1099.
- [3] Lucas, G. E., 1983, "The Development of Small Specimen Mechanical Tests Techniques," *J. Nucl. Mater.*, **117**, pp. 327–339.
- [4] Hyde, T. H., Sun, W., and Williams, J. A., 2007, "Requirements for and Use of a Miniature Test Specimens to Provide Mechanical and Creep Properties of Materials: A Review," *Int. Mater. Rev.*, **52**(4), pp. 213–255.
- [5] Lucas, G. E., 1990, "Review of Small Specimen Test Techniques for Irradiation Testing," *Metall. Trans. A*, **21**(5), pp. 1105–1119.
- [6] Lucas, G. E., Odette, G. R., Matsui, H., Moslang, A., Spatig, P., Rensman, J., and Yamamoto, T., 2007, "The Role of Small Specimen Tests Technology in Fusion Materials Development," *J. Nucl. Mater.*, **367–370**(Part B), pp. 1549–1556.
- [7] Wakai, E., Nogami, S., Kasada, R., Kimura, A., Kurishita, H., Saito, M., Ito, Y., Takada, F., Nakamura, K., Molla, J., and Garin, P., 2011, "Small Specimen Test Technology and Methodology of IFMIF/EVEDA and the Further Subjects," *J. Nucl. Mater.*, **417**(1–3), pp. 1325–1330.
- [8] Razali, A. R., and Qin, Y., 2013, "A Review on Micro-Manufacturing, Micro-Forming and Their Key Issues," *Procedia Eng.*, **53**, pp. 665–672.
- [9] Eichehueler, B., 2007, "Microforming at Elevated Temperature Forming and Material Behavior," *Int. J. Adv. Manuf. Technol.*, **33**(1), pp. 119–124.
- [10] Geiger, M., Kleiner, M., Eckstein, R., Tiesler, N., and Engel, U., 2001, "Microforming," *CIRP Ann.—Manuf. Technol.*, **50**(2), pp. 445–462.
- [11] Fu, M. W., and Chan, W. L., 2014, *Micro-Scales Products Developments Via Microforming* (Springer Series in Advanced Manufacturing), Springer-Verlag, London, pp. 9–41.
- [12] Czoboly, E., Gillemot, F., and Oszwald, F., 1998, "Small Specimen Testing Applied at Surveillance Extension," *Small Specimen Test Techniques*, ASTM, Philadelphia, PA, pp. 163–172, Standard No. STP 1329.
- [13] Hankin, G. L., Toloczko, M. B., Hamilton, M. L., Garner, F. A., and Faulkner, R. G., 1998, "Validation of the Shear Punch-Tensile Correlation Technique Using Irradiated Materials," *J. Nucl. Mater.*, **258–263**(Part 2), pp. 1651–1656.
- [14] Nedoushan, R. J., Farzin, M., and Banabic, D., 2014, "Simulation of Hot Forming Process: Using Cost Effective Micro-Structural Constitutive Models," *Int. J. Mech. Sci.*, **85**, pp. 196–204.
- [15] Haque, M. A., and Saif, M. T., 2004, "Deformation Mechanisms in Free-Standing Nanoscale Thin Films: A Quantitative In Situ Transmission Electron Microscope Study," *PNAS*, **101**(17), pp. 6335–6340.
- [16] Ma, X., and Shi, H., 2014, "In Situ SEM Studies of the Low Cycle Fatigue Behavior of DZ4 Superalloy at Elevated Temperature: Effect of Partial Recrystallization," *Int. J. Fatigue*, **61**, pp. 255–263.
- [17] Guo, E. Y., Wang, M. Y., Jing, T., and Chawla, N., 2013, "Temperature-Dependent Mechanical Properties of an Austenitic–Ferritic Stainless Steel Studied by In Situ Tensile Loading in a Scanning Electron Microscope," *Mater. Sci. Eng. A*, **580**, pp. 159–168.
- [18] Petreneč, M., Polák, J., Šamorík, M., Dluhoš, J., and Obrtlík, K., 2014, "In-Situ Study of the Mechanisms of High Temperature Damage in Elastic–Plastic Cyclic Loading of Nickel Superalloy," *Adv. Mater. Res.*, **891–892**, pp. 530–535.
- [19] Petreneč, M., Polák, J., Šamorík, M., Dluhoš, J., and Obrtlík, K., 2014, "In-Situ High Temperature Low Cycle Fatigue Study of Surface Topography Evolution in Nickel Superalloy," 23rd International Conference on Metallurgy and Materials (METAL), Brno, Czech Republic, May 21–23.
- [20] Callaghan, M. D., Humphries, S. R., Law, M., Li, H., and Yeung, W. Y., 2010, "Evaluation of High Temperature Fatigue Behavior of P22 by Miniature Specimen Testing," *Mater. Sci. Forum*, **638–642**, pp. 3937–3942.
- [21] Kohyama, A., Sato, S., and Hamada, K., 1993, "An Automated Tensile Machine for Small Specimens Heavily Neutron Irradiated in FFTF/MOTA," *Small Specimen Test Techniques Applied to Nuclear Reactor Vessels Thermal Annealing and Plant Life Extension*, W. R. Corwin, F. M. Haggag, and W. L. Server, eds., American Society for Testing and Materials, Philadelphia, PA, pp. 356–367.
- [22] Hannula, S. P., Wanagel, J., and Li, C. Y., 1986, "A Miniaturized Mechanical Testing System for Small-Scale Specimen Testing," *The Use of Small-Scale Specimens for Testing Irradiated Material*, W. R. Corwin and G. E. Lucas, eds., American Society for Testing and Materials, Philadelphia, PA, pp. 233–251.
- [23] Quested, T. E., 2000, "Finite Element Analysis of Miniaturised Electro Thermo Mechanical Testing System," NPL Report CMMT (A) No. 291.
- [24] Ho, C. Y., and Chu, T. K., 1977, "Electric Resistivity and Thermal Conductivity of Nine Selected AISI Stainless Steels," U.S. Defense Technical Information Center, Defense Logistics Agency, Alexandria, VA, CINDAS Report No. 45, pp. 5–30.