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TEST SYSTEM FOR ELEVATED TEMPERATURE CHARACTERIZATION OF THIN METALLIC FOILS (PREPRINT)

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14. ABSTRACT
The need to accurately determine the mechanical properties of thin metallic foil materials has grown in recent years as the emphasis on hypersonic vehicles and access to space has also increased. These foils, with thicknesses ranging from 75-250 μm, are used in the fabrication of metallic sandwich panels consisting of honeycomb structures, which are used for thermal protection of numerous space and hypersonic vehicles. Typically, these panels are designed with material properties determined using sheet data (thickness ≥ 1000 μm) due to the lack of data available for the foil product form. Limited data are available in the open literature for foil gage metals. El-Soudani reported the properties of foil gage (254 μm) specially processed Ti-6242S up to 593ºC. Zupan used microsamples to characterize the tensile behavior of thin gage Gamma TiAl. Moreau et al. used a four-point bend testing configuration to study the effect of thickness on yield strength of nickel foils at room temperature. Lara-Curzio et al. studied the effect of thermal exposure on the tensile behavior of a Ni-base superalloy, Haynes 230®.

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Test System for Elevated Temperature Characterization of Thin Metallic Foils

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OVERVIEW

The need to accurately determine the mechanical properties of thin metallic foil materials has grown in recent years as the emphasis on hypersonic vehicles and access to space has also increased [1-5]. These foils, with thicknesses ranging from 75-250 μm, are used in the fabrication of metallic sandwich panels consisting of honeycomb structures, which are used for thermal protection of numerous space and hypersonic vehicles [3-5]. Typically, these panels are designed with material properties determined using sheet data (thickness ≥ 1000 μm) due to the lack of data available for the foil product form. Limited data are available in the open literature for foil gage metals. El-Soudani [5] reported the properties of foil gage (254 μm) specially processed Ti-6242S up to 593ºC. Zupan [6] used microsamples to characterize the tensile behavior of thin gage Gamma TiAl. Moreau et al. [7] used a four-point bend testing configuration to study the effect of thickness on yield strength of nickel foils at room temperature. Lara-Curzio et al. [8] studied the effect of thermal exposure on the tensile behavior of a Ni-base superalloy, Haynes 230®. Miniature specimens with 89-102 μm thickness were tested at room temperature following exposure to elevated temperatures. In these tests, Lara-Curzio et al. [8] did not measure the strain directly. The cross-head displacement was used to deduce the specimen displacement. Recently, Liu and Holmes [9] developed a test system to study the crack growth behavior of Ni-base superalloy foils (76 μm) up to 760ºC. These tests also did not involve direct displacement measurements on the specimens.

Ni-base superalloy foil gage materials are being targeted for applications up to 1100ºC. To achieve an optimized system design, standard mechanical behavior data on these materials are needed under a range of loading conditions such as tensile, creep and fatigue at representative temperatures. These tests require direct measurements of displacements on specimens.

In order to meet this need, a test system has been developed as part of a comprehensive in-house program to advance state-of-the-art testing capabilities for thin foils and very thin sheets. The test system was developed using a conventional MTS hydraulic load frame outfitted with specialized capabilities, and was designed for determining materials properties on a macro-scale. Specimen thicknesses used in this study range from 125-500 μm, with specimen lengths on the order of 150 mm. This paper outlines the developmental process, including unique challenges, as well as the system validation and some preliminary data.

MATERIAL

The initial material of interest for the Metallic Thermal Protection System (TPS) program was the nickel-base superalloy Haynes 230®. Haynes 230® is a solid solution strengthened alloy based on the Ni-Cr-Mo-W system. It is widely used in combustors, ducting, hot gas housings, stator casings and other static components used in land-based turbines. It is known for its high temperature strength, excellent oxidation resistance up to about 1150C and good long-term thermal stability [10].

The material was received in the form of three, twenty-five pound, 31.75 cm (2.5”) wide coils rolled to thicknesses of 127, 254, and 508 μm (5, 10, and 20 mils, respectively). All of the material was rolled from the same starting ingot, and a comparison of the measured alloy composition to the baseline composition is given in Table 1. The rolling operations were performed by the Elgiloy Corporation of Elgin, IL. Detailed characterization of the Haynes 230® foil can be found elsewhere [11].

Table 1: Comparison of the baseline (nominal) and actual alloy compositions.

	Ni	Cr	W	Mo	Fe	Co	Mn	Si	Al	C	La	B	Heat Code
baseline	57[a]	22	14	2	<3*	<5*	0.5	0.4	0.3	0.1	0.02	<0.015*	
All material	bal	22.546	14.178	1.299	1.245	0.224	0.544	0.376	0.381	0.108	0.015	0.002	830547842
	*Maximum	[a]As balance											

SPECIMEN DESIGN AND PREPARATION

The specimens for the macro-test system are flat dogbones with a total length of 152 mm and gage section minimum width of 8.50 mm, as shown in Figure 1. This varies slightly from the design designated in ASTM E345-93, “Standard Test Methods of Tension Testing of Metallic Foil [12]. The ASTM standard designates a similar dogbone specimen design, although with slightly different dimensions. Some of the differences include minimum dimensions of 200 mm, 50 mm, and 19mm for the specimen length, gage length, and fillet radius. When finite element calculations were performed on varying specimen sizes that would conform to ASTM standards, it was found that fairly large stress concentrations could exist at the edge of the gage section, resulting in stresses 18% higher than the nominal gage section stress. The radius of curvature was therefore increased to 50 mm, reducing the maximum stresses to 4.7% above the nominal gage section stress. In addition, the overall specimen and gage lengths were decreased slightly, which enables specimens to be cut from smaller sections of foil material.

Due to the specimen thickness, it is not feasible to machine them directly. Instead, blanks are cut to length, stacked and secured between 12.7 mm steel plates (Figure 2). The number of specimens machined at one time varies based on foil thickness, but is generally between 12 and 20. A wire EDM is then used to machine the stack of specimens to final dimensions. Before being removed from the steel clamping plates, the machined specimens are polished to remove burrs and machining marks from the specimen edges. Polishing consists of a three step process using 240, 400, and finally 600 grit paper, resulting in a 0.8 μm finish. The specimens are then removed from the stack and inspected under a microscope to verify that the edges are smooth and that there are no surface scratches or creases, per ASTM recommendation.

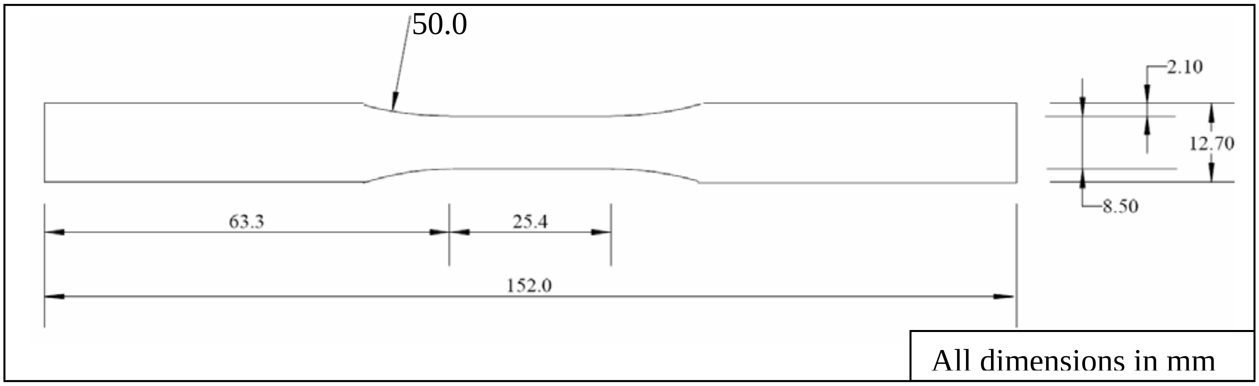
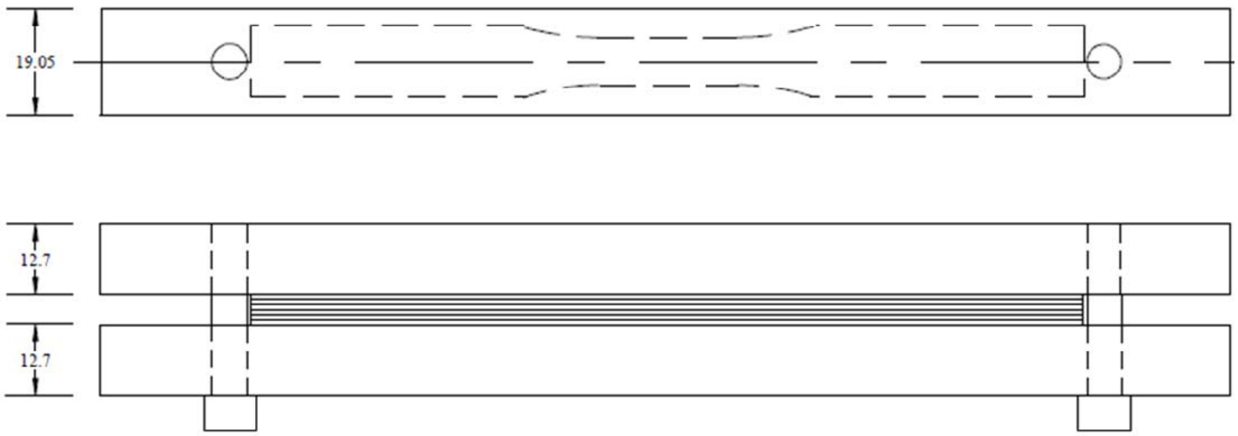


Figure 1: Specimen design



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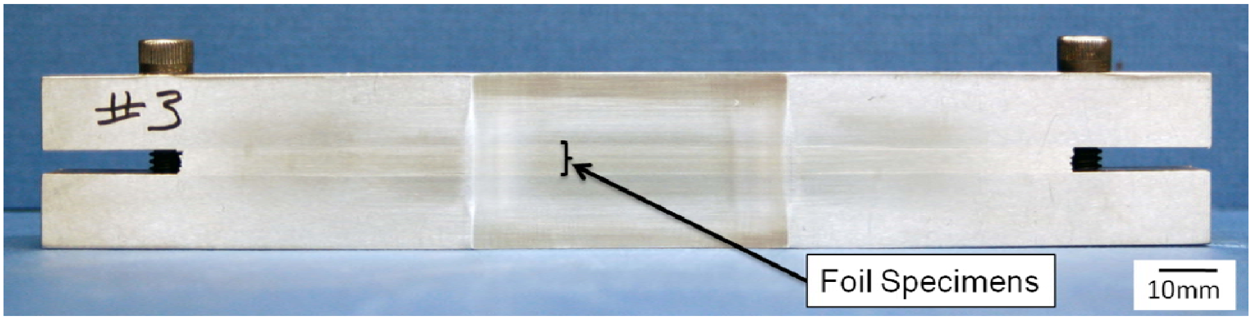

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Figure 2: a) Schematic of specimen blanks held by steel plates (all dimensions in mm), and b) Edge view of Haynes 230® specimens after machining and polishing.

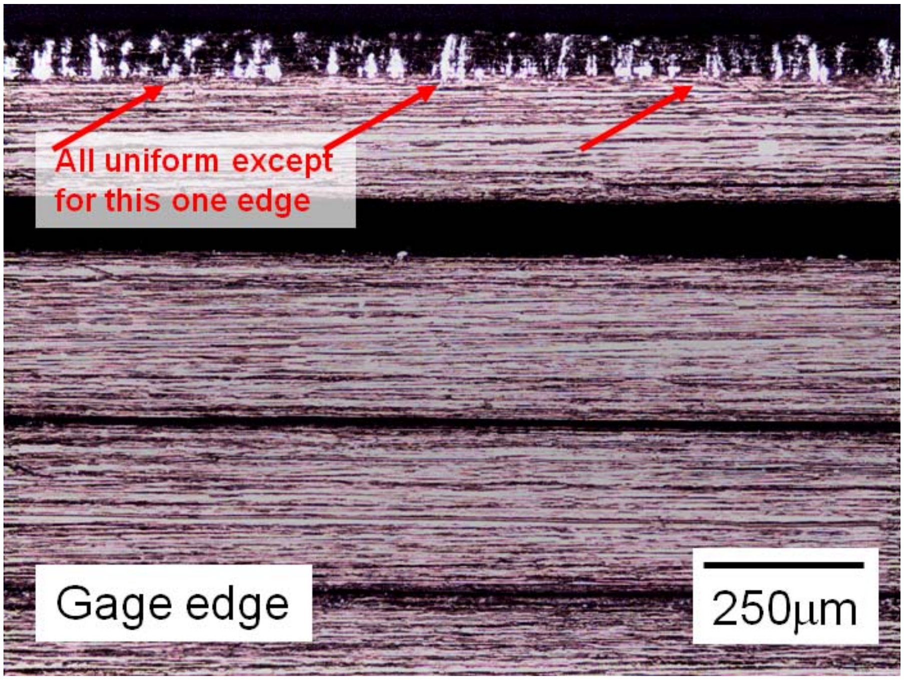

Figure 3: Specimen gage section edges of 254 μm foil specimens shown under optical microscope. One edge of top specimen shows the original machining marks.

SYSTEM DESIGN

The macro-test system was designed to determine mechanical properties of thin foils (5-30 μm) using specimens of conventional length (152 mm) and gage width (8.5 mm). The property values determined will be directly applicable to industry applications where thin foil materials are used over a large area, such as in thermal protection systems (TPS). A conventional MTS hydraulic load frame outfitted with a digital controller serves as the backbone of the system. A horizontal load frame, which minimizes the chimney effect and was designed for high temperature testing [13, 14], was chosen for this system. However, very thin samples present several unique challenges which had to be overcome. The following sections describe the modifications that have been made to accommodate the unique requirements.

Specimen Alignment

The test system is equipped with 10,000 psi hydraulic grips attached to the horizontal load frame. Two fixtures were designed for the test system in order to minimize specimen misalignment and bending in the grips. The first, an alignment fixture designed with two micrometers attached to a small steel plate, was designed to ensure that the specimen was properly aligned with the grips and load train. The fixture, shown in Figure 4, is used each time a specimen is loaded in the test frame. The second fixture is used to eliminate out of plane motion by the grips. This alignment jig, shown in Figure 5, is designed to fit on top of the grip assembly and is also used each time a specimen is put installed. It is then removed prior to the start of the test.

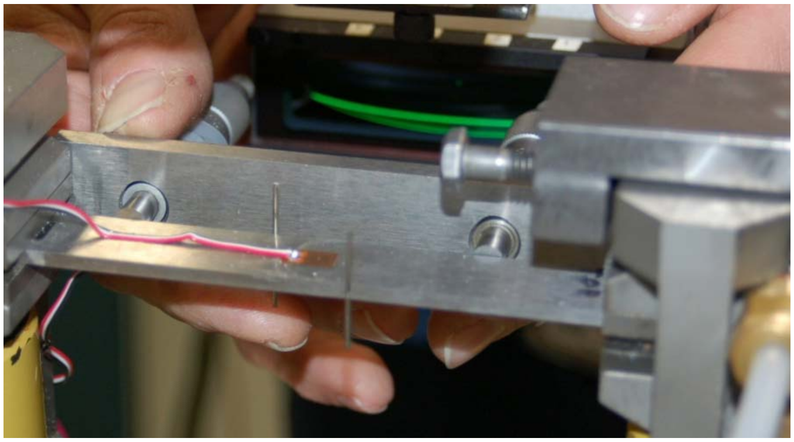

Figure 4: Specimen Alignment Fixture

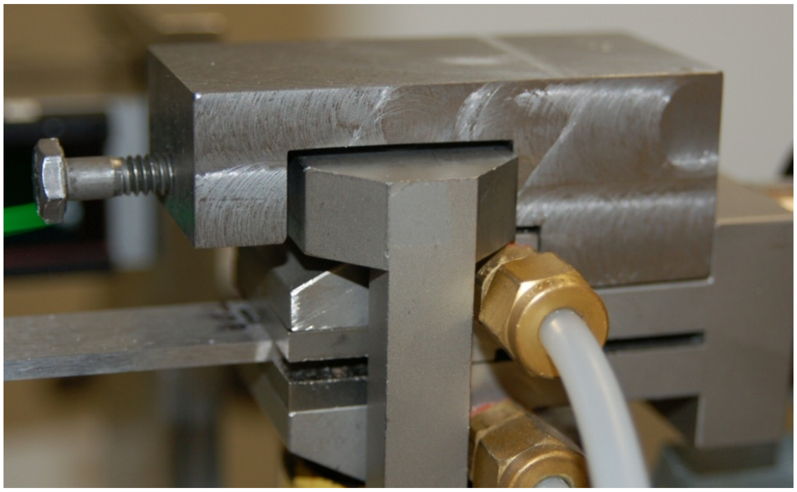

Figure 5: Grip Alignment Fixture

Non-Contact Displacement Measurement

Perhaps the most critical, and most difficult, aspect of testing thin foils involves the displacement measurement of the specimens within the gage section. Due in part to the unique horizontal arrangement of the test system, the displacement in the gage section is measured using two vertical rods hanging from the specimen. These rods, or flags, rest on top of the specimen at a point, as well as along the side of the specimen (Figure 6). After a specimen has been loaded in the test frame, the flags are placed on the gage section using an alignment jig. In this way, the flag position relative to each other remains consistent from test to test at approximately 18 mm. The flags, made from a Ni base single crystal superalloy, allow the direct measurement of the displacement in the gage section between the two flags. Originally the system was designed using a laser micrometer to measure the flag movements, and therefore the displacement. The laser micrometer offers advantages such as good stability and exceptional accuracy (± 0.5 μm). However, it is limited by the data collection speed of 100 Hz. This slow speed causes problems during tensile tests run within ASTM standards by limiting the amount of data points collected.

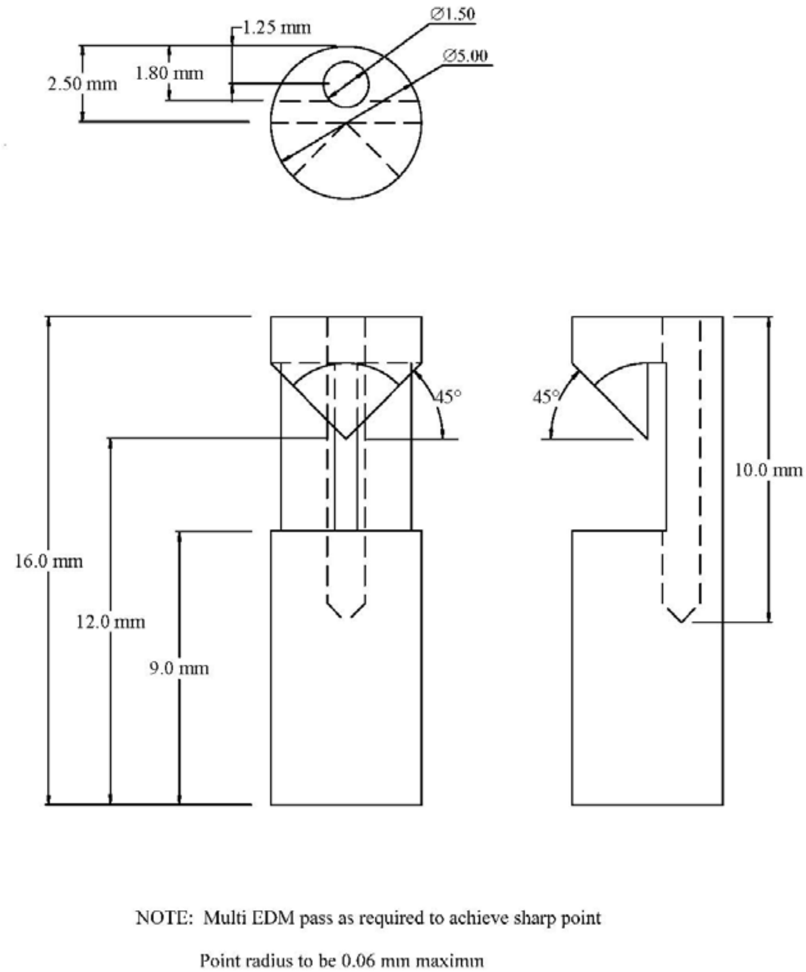

Figure 6: Schematic of flag made from single crystal superalloy.

This becomes an even bigger issue when testing at elevated temperatures where creep effects will occur very quickly, reducing the allowable time for test completion.

Due to the limitations of the laser micrometer system, a new digital optical micrometer was installed. This system uses a CCD array to provide continuous displacement data at over 2 kHz, as opposed to the linear scan of the laser system at 100 Hz. The digital system also provides two channels of data, which can be formatted independently to record data at using various averaging and input rates. This is very useful for separating high resolution data in the linear region from more periodic data over a wider displacement area during a complete tensile test.

Elevated Temperature Furnace

One potential use for thin foil metallic materials is as components of thermal protection systems. In order to determine the applicability of a material for such an application, material properties at elevated temperatures are required. Therefore, a significant goal of this project is to have the ability to test thin foil samples at elevated temperatures. Because of the unique test setup and displacement measuring system, an acceptable commercial-off-the-shelf (COTS) furnace was not available. Instead, a furnace was designed and built in-house. The furnace was specially designed with the following features: viewing ports on the front and back of the furnace, three zone heating, limited size to fit within the test system confines, and a temperature capability exceeding 1200ºC.

The furnace is constructed and mounted as two separate pieces. The top and bottom portions are both aligned and mounted on motorized translational stages, which allow them to be raised and lowered independently. This allows the furnace to be easily accessed for specimen loading, and easily closed for elevated temperature testing. The viewing ports within the furnace allow the optical micrometer to measure the displacement of the flags throughout each test. Multi-zone heating is accomplished using silicon nitride igniters, which have higher temperature capability and increased durability over silicon carbide igniters. Six igniters are divided into three zones, with each zone controlled independently. This allows for very stable, uniform temperatures across the specimen gage length, as shown in Figure 7. Initial testing of the furnace has verified temperature capability of 1200ºC. The bottom half of the furnace assembly can be seen in Figure 8. Included in the picture are the three silicon nitride ingnitors used to control the three temperature zones, a specimen, and the single crystal flags used to measure the gage section displacement. Figure 9 shows the complete test setup as seen during testing. The three wires coming from the furnace are from the thermocouples used to monitor and control heating during testing. Also shown in the picture are the optical micrometer, water cooled grips, and furnace housing. One window in the furnace, used by the optical micrometer when measuring the specimen displacement, is shown in Figure 10.

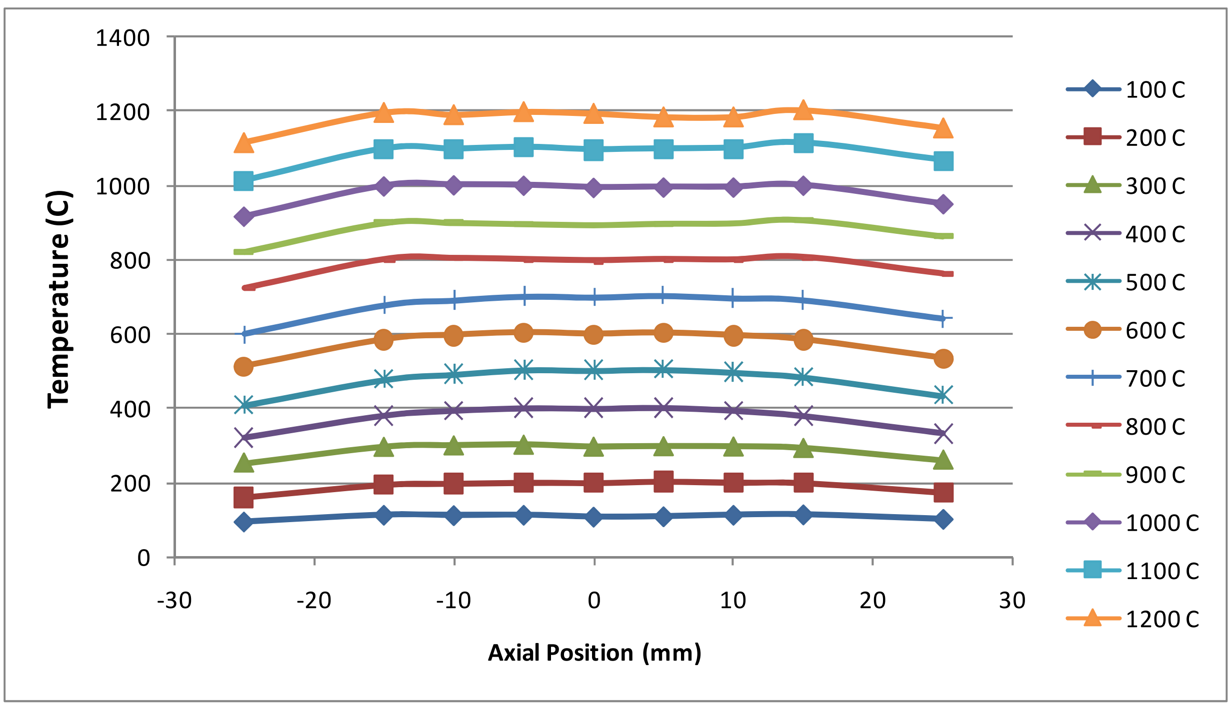

Figure 7: Temperature profile obtained in the furnace.

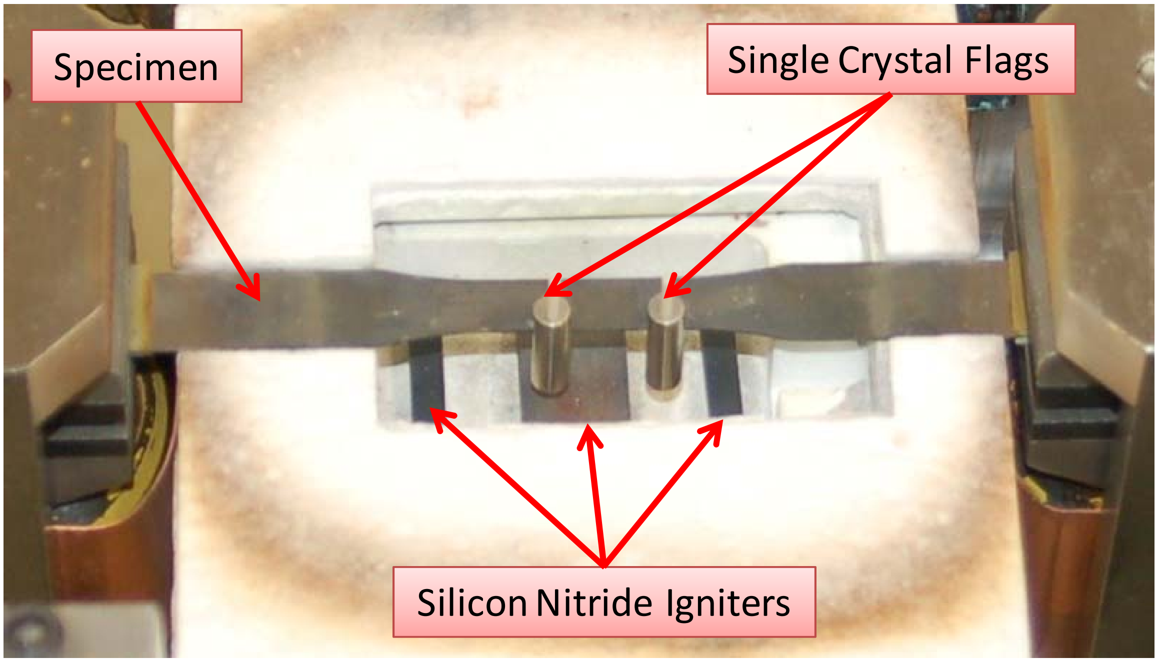

Figure 8: Lower portion of furnace including specimen and displacement flags.

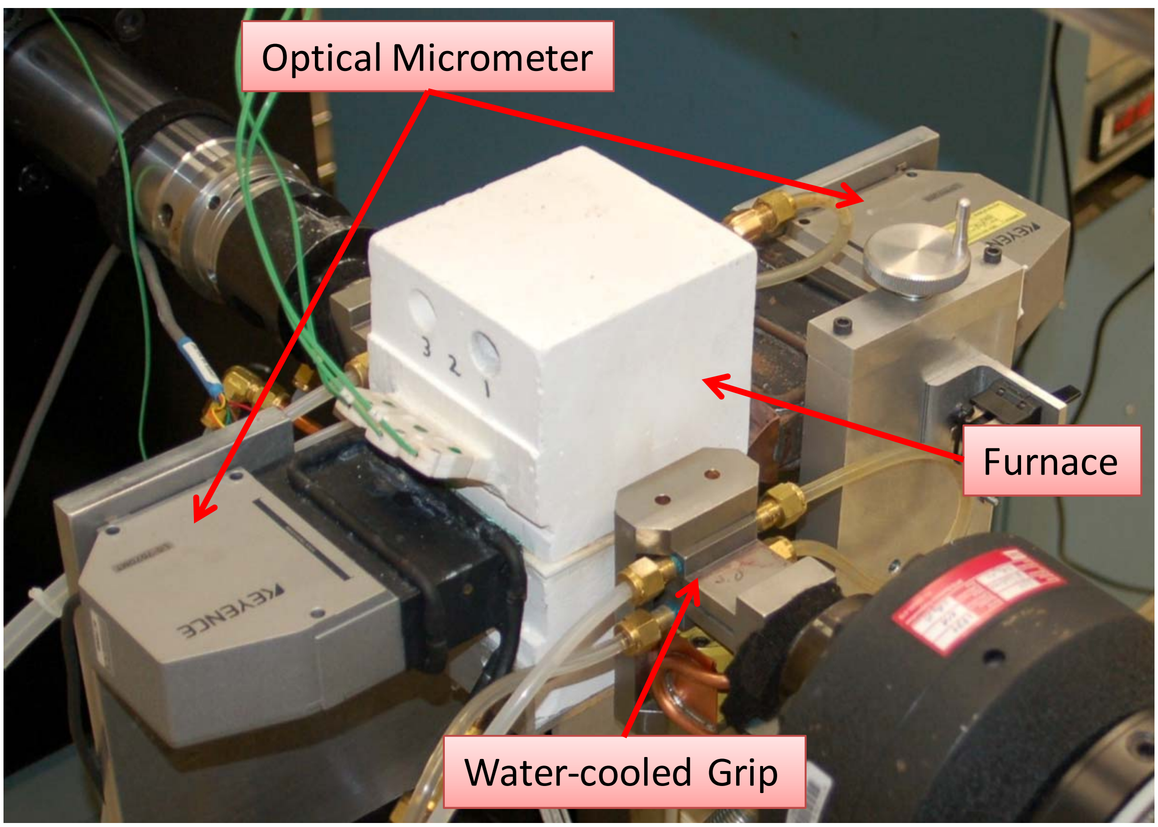

Figure 9: Test setup.

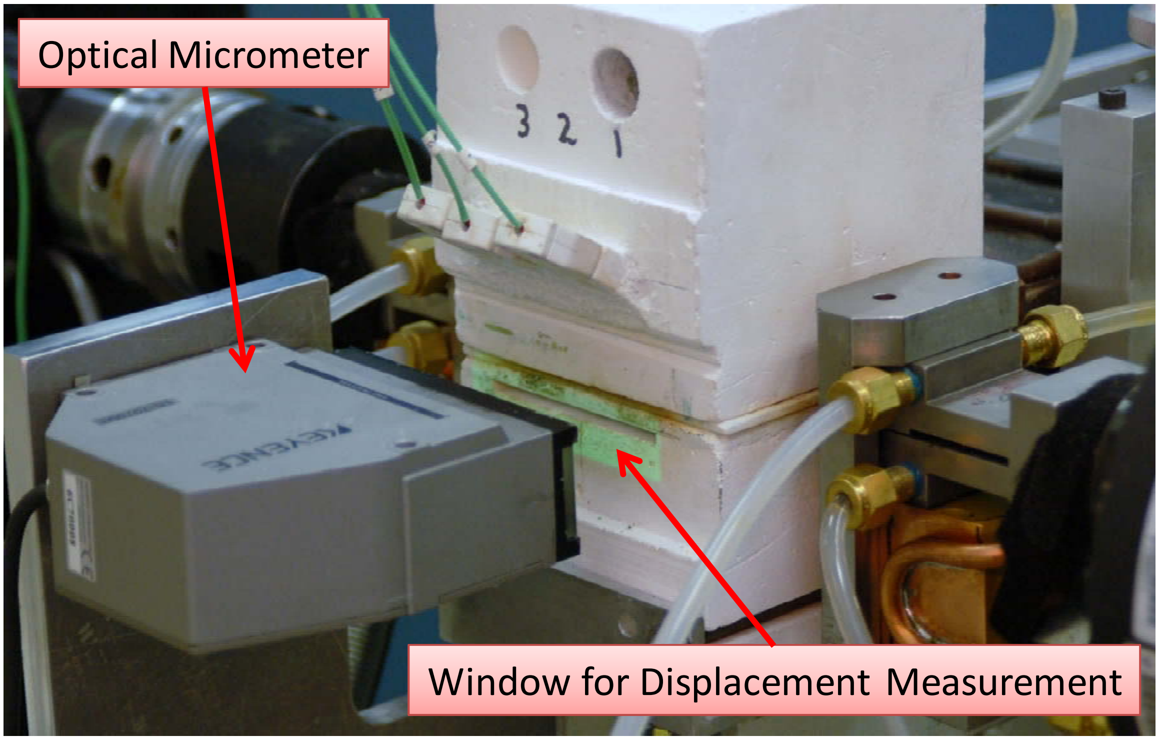

Figure 10: Close up of furnace showing window used for displacement measurement.

Test Procedure

A large amount of effort has been spent determining a test procedure which will result in accurate and repeatable data. After careful examination consideration, the following test procedure has been determined, and is now used for all testing.

Room Temperature Tensile

1) Strain gages attached to top and bottom of gage section
2) Specimen loaded into system
3) Displacement flags placed on specimen, distance between flags determined
4) Modulus check at 5 MPa/s to verify system stability and compare displacement measurements with strain gage data (2-3 times)
5) Modulus check at stress rate necessary to achieve 0.030 mm/s loading rate (2 times)
6) Run tensile test under displacement control at 0.030 mm/s

Elevated Temperature Tensile

1) – 5) Repeat room temperature procedure
6) Remove strain gage leads

7) Close furnace, and heat specimen to desired test temperature
8) Allow furnace and specimen to stabilize at proper temperature (~30 minutes)
9) Modulus check at stress rate necessary to achieve 0.030 mm/s loading rate (2 times)
10) Run tensile test under displacement control at 0.030 mm/s

The loading rates were determined by first testing rates taken from the applicable ASTM standard, and then adjusting as necessary. The initial room temperature modulus checks, performed at 5 MPa/s, are within the range designated by ASTM E08 (1.15-11.5 MPa/s) concerning room temperature tensile testing as well as ASTM E345 (1.2-12 MPa/s) on room temperature tensile testing of metallic foils. ASTM E21 designates a strain rate of 0.005±0.002 m/m/min for yield properties. This corresponds to a displacement rate on the order of 0.002 mm/s for specimens used in this study. For very high temperature applications such as thermal protection systems, the materials of interest will begin to creep at such slow loading rates. The loading rate chosen for this work was 0.030 mm/s, which corresponds to a strain rate of approximately 0.06 m/m/min. This rate falls within the region designated by E08 for room temperature tensile testing in the plastic regime (0.05-0.5 m/m/min).

Results and Discussion

Extensive testing of the system components was performed to determine their operational capabilities. These tests were often performed using steel sheet material (0.5-1.5 mm thick) to eliminate issues caused by the thin foils. Recently this phase of testing was completed, and validation tests to verify the accuracy of the noncontact displacement system at both room and elevated temperature were performed using the Haynes 230® specimens with a thickness of 508 μm.

The validation of this system, using various combinations of displacement range, averaging, and scan rate, has been completed. The results from a room temperature tensile test, using the test procedure detailed above, are shown in Figure 11. At room temperature, the optical micrometer shows very good agreement with the strain gage data over the whole range of data, which includes all data up to strain gage failure. The modulus of elasticity determined using the optical system also closely matches that determined using strain gage data as well as the published values for Haynes 230®. In addition, while strain gages will fail at strains on the order of 2%, the displacement system using the optical micrometer can record large displacements, allowing strain measurements greater than 50%. This results in a much more accurate picture of the true material response than could be determined with strain gages and grip displacements.

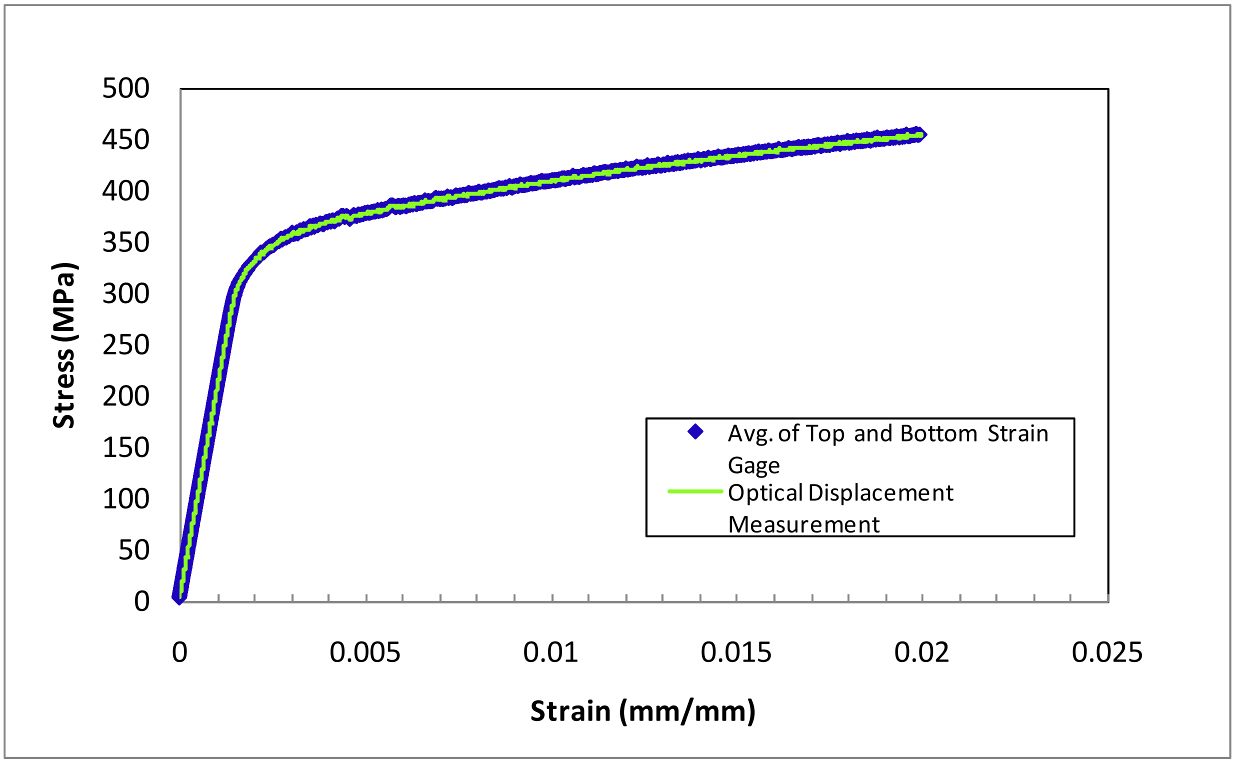

Figure 11: Comparison of strain gage data with strain determined using optical displacement measurements.

Following the room temperature validation, testing was performed to validate the non-contact displacement system at elevated temperatures. This involved measuring the displacement of the flags while looking through the port windows in the furnace. Comparing data directly to strain gage values was not an option due to the high temperatures. Therefore, validation of the displacement measurement system was done at a range of temperatures by comparing the thermal strain determined from measured displacements with those calculated using published coefficient of thermal expansion (CTE) data for Haynes 230®. A specimen was loaded and held at a very low stress (5 MPa) at room temperature and the displacement in the gage section was recorded. The specimen was then heated slowly to 100ºC and a new displacement reading was taken after the furnace had reached a uniform temperature. This was repeated in 100ºC increments. Published CTE data as a function of temperature [10] was then used to determine the expected displacement and strain at each temperature. The thermal strain determined with measured displacemensts and published CTE values were then plotted as a function of temperature (Figure 12). The predicted values agree well with the data validating the elevated temperature displacement measurement capability of the non-contact system developed in this study.

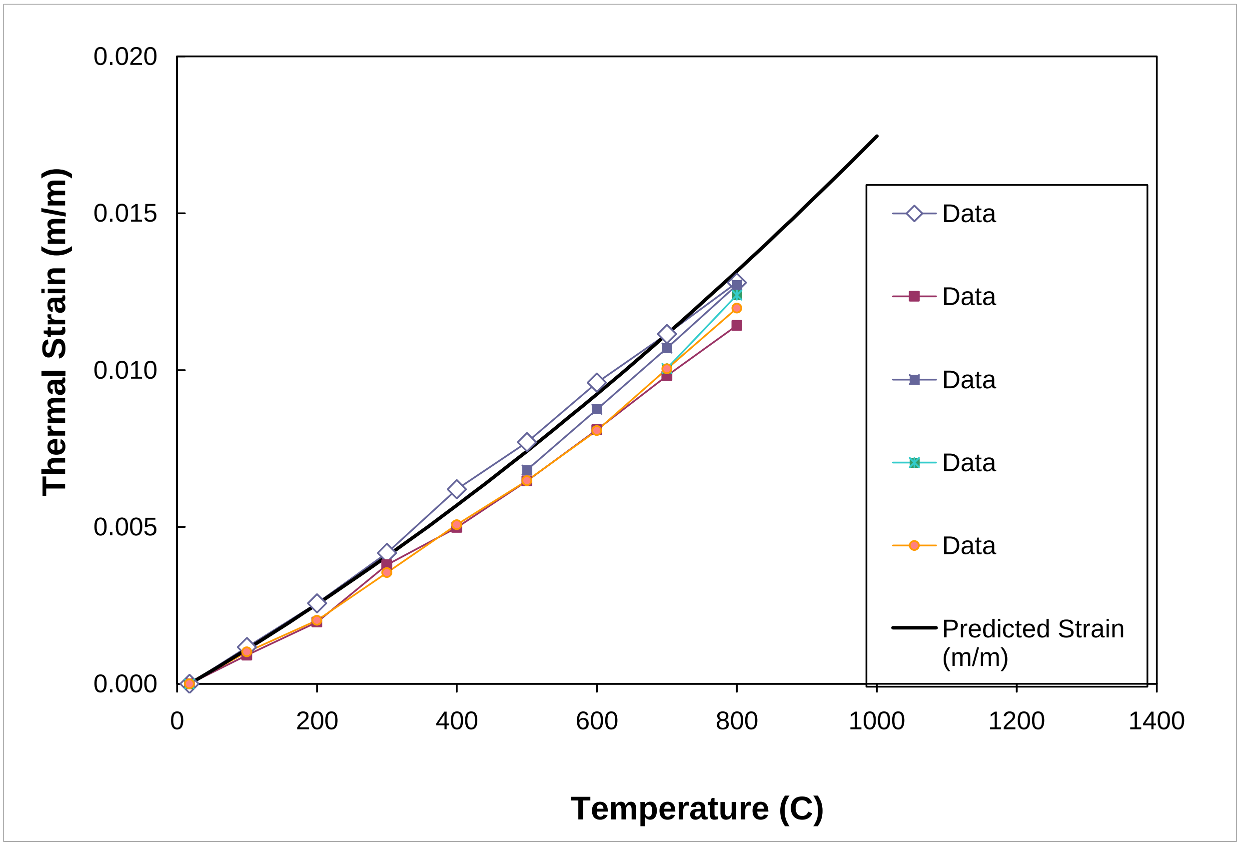

Figure 12: Measured and calculated thermal strain for Haynes 230® foil as a function of temperature.

In addition to closely matching strain gage data during a short duration tensile test, it is important that the optical displacement system stable measurements over long durations. Therefore, the stability of the optical system was verified by measuring displacement data over a 24 hour time period at room and elevated temperature.

Upon completion of the validation testing, tensile tests were performed using Haynes 230® foil with a thickness of 127 μm (5 mil). The results of two room temperature tensile tests are shown in Figure 13. Also included in the figure are the modulus (199 GPa) and 0.2% offset yield strength (450 MPa), which are slightly different than published [10] plate data for Haynes 230® (211 GPa and 422 MPa, respectively). These differences are not unexpected due to the extra processing steps that are required to produce the product as a foil. Additional work to investigate these differences using various thicknesses of plate and foil will be performed in the near future.

Figure 14 shows the results of tensile tests performed at 816ºC (1500 F). As was the case for the room temperature results, the values vary somewhat from published plate data. As was the case at room temperature, the modulus value of 153 GPa is slightly lower than those published [10] for the plate at 760ºC (171 GPa) and 871ºC (164 GPa). The yield stress is harder to determine based on the serrated yielding that occurs at the elevated temperature, but is close to 300 MPa. This falls within the range published [10] for 760ºC (323 MPa) and 871ºC (234 MPa), although slightly higher than expected based on simple interpolation (similar to the room temperature results).

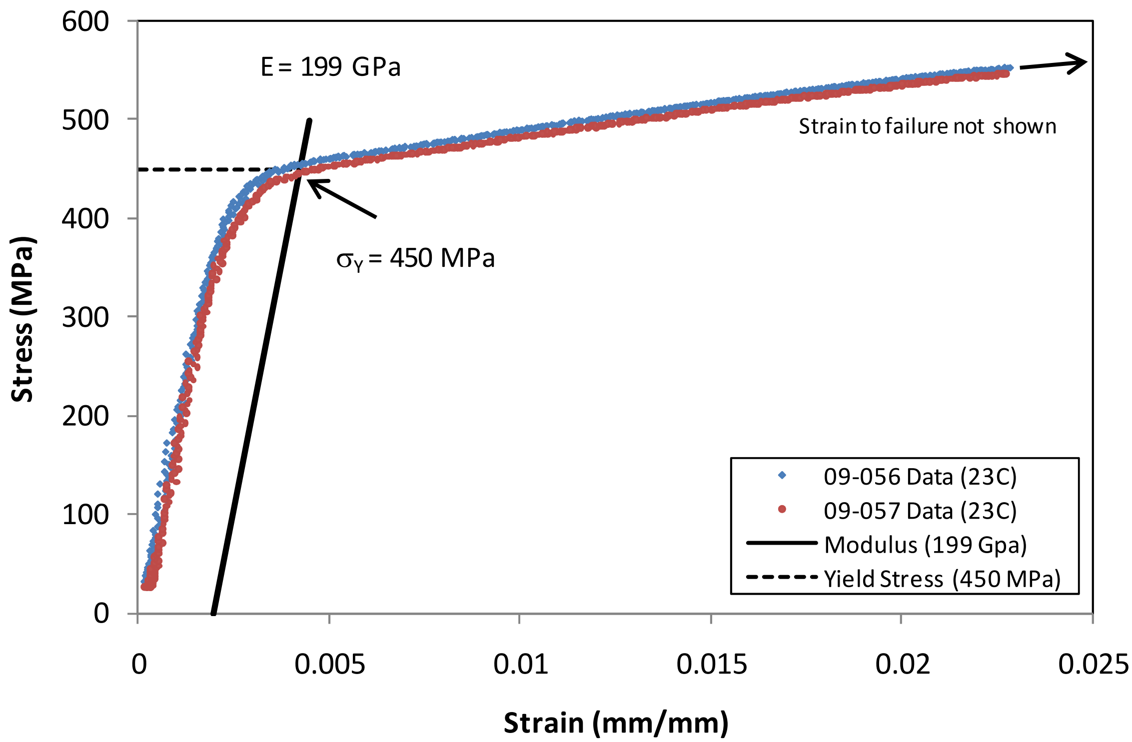

Figure 13: Room temperature tensile results for 127 μm Haynes 230® foil.

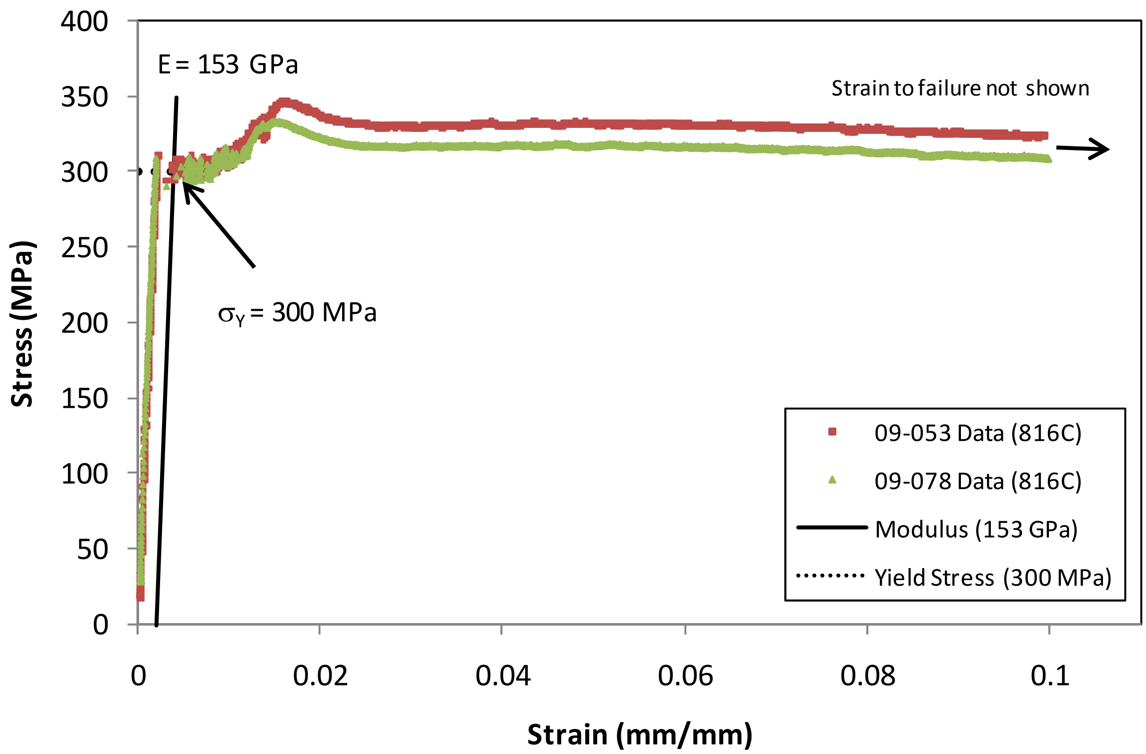

Figure 14: Elevated temperature (816 C/1500 F) tensile results for 127 μm Haynes 230® foil.

Summary

A new, state-of-the-art test system for thin foil materials (125-508 μm) has been developed based on a conventional servohydraulic load frame. It includes a novel approach for measuring gage section displacements, as well as a unique furnace design that enables non-contact displacement measurement at elevated temperatures, up to 816ºC. The test system has the ability to accurately measure displacements in the gage section of conventional specimens at elevated temperatures out to very large strains (> 40%). This test system will allow more direct mechanical property measurements on the materials used to fabricate current and future thermal protection systems, as well as other high temperature applications. Although new in design, all aspects of the test system are commercial, off-the-shelf items, which will allow development of a similar capability in other laboratories.

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