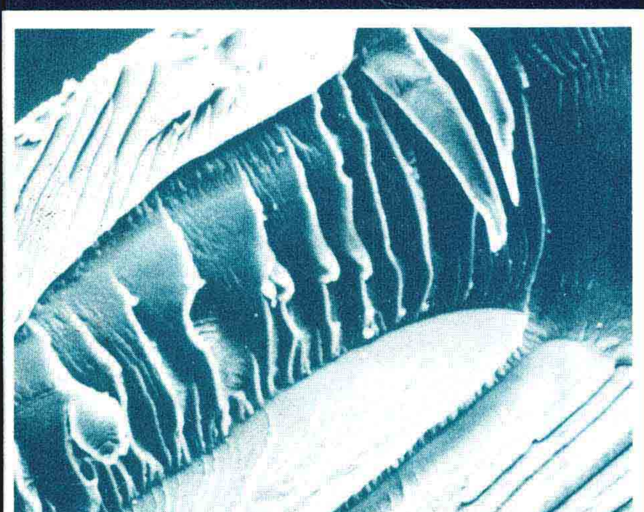
Test Methods and Design Allowables for Fibrous Composites

Second Volume



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editor

ASTM

STP 1003

STP 1003

Test Methods and Design Allowables for Fibrous Composites: 2nd Volume

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ASTM
1916 Race Street
Philadelphia, PA 19103

Library of Congress Cataloging-in-Publication Data

Test methods and design allowables for fibrous composites / Christos C. Chamis, editor.

"2nd volume"
"Test methods and design allowables for fiber composites: second symposium was held at Phoenix, AZ, on 3-4 Nov. 1986. ASTM Committee D-30 on High Modulus Fibers and Their Composites sponsored by the symposium."--Fwd.
Includes bibliographies and index.
ISBN 0-8031-1196-7
1. Fibrous composites--Congresses. I. Chamis, C. C. (Christos C.) II. ASTM Committee D-30 on High Modulus Fibers and Their Composites. III. Series: ASTM special technical publication ; 1003.
TA418.9.C6T43 1989
620.1'18--dc19 88-38493
CIP

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The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution of time and effort on behalf of ASTM.

Printed in Baltimore, MD
February 1989

Foreword

Test Methods and Design Allowables for Fiber Composites: Second Symposium was held at Phoenix, AZ, on 3-4 Nov. 1986. ASTM Committee D-30 on High Modulus Fibers and Their Composites sponsored the symposium. Christos C. Chamis, NASA Lewis Research Center, served as chairperson of the symposium and is editor of the resulting publication.

Overview

The composites structures community recognizes that fiber composites offer a multitude of desirable properties to meet diverse and competing design requirements in a cost-effective manner. This multitude of desirable properties, however, requires special test methods to quantify material properties for design. It also requires well-defined procedures in selecting and setting design allowables for composites. Over the past 25 years researchers have continuously sought test methods to measure composite material properties with an acceptable degree of repeatability. At the same time, designers have sought well-defined procedures to select and establish design allowables that are compatible with current practice margins. Special test methods to measure composite properties and procedures for setting design allowables go through a long period of peer evaluation and critique before their adaptation on a general consensus basis.

In order to shorten the peer evaluation and critique period, a specialists symposium was organized 2 and 3 Oct. 1979, in Dearborn, MI, with the objective to provide a forum for the discussion of (1) special test methods for setting design allowables and reporting results; (2) selecting and establishing design allowables, (3) promoting an understanding of the procedures for establishing design allowables, and (4) developing new test methods streamlined for setting design allowables that will eventually lead to improved methodology for reliable composite structures design. Papers presented in that symposium are included in ASTM *STP 734, Test Methods and Design Allowables for Fibrous Composites,* which was published in 1981.

During the past ten years, new composites have emerged, especially for high-temperature and hostile environment applications. These new applications require new test methods. The evolution of new test methods and the improvement of existing ones constituted a ripe time to have a second specialist symposium on "Test Methods and Design Allowables for Fiber Composites," which was held on 3–4 Nov. 1986, Phoenix, AZ, and which focused on these recent developments. The papers presented in the second symposium are grouped into three sessions: (1) Extreme/Hostile Environment Testing, (2) Establishing Design Allowables, and (3) Property/Behavior Specific Testing. The papers presented in these sessions are included as three respective sections in this Special Technical Publication (STP). A brief description of each section follows: Section I on Extreme/Hostile Environment Testing contains six papers on testing under conditions of extreme temperatures, sand erosion, abrasive wear, and other environmental variables. Section II on Establishing Design Allowables presents five papers that review current practices and statistical methods. Section III on Property/Behavior Specific Testing contains six papers on delamination, in-plane shear, torsional failure, elastic-plastic stress, and fatigue testing.

The papers in each section provide a valuable source as to where the advances and emphasis were at the time of the second symposium with respect to special testing and procedures for selecting design allowables. In addition, the papers provide a good perspective, with suitable references, of the test methods and procedures for setting design allowables that have been proposed, or are used, including ranges of applications and limitations.

Furthermore the papers in each major area offer specific recommendations for future research, which will lead to improved test methods and procedures for establishing design allowables for high-temperature composites and for the use of composites in hostile environments. Lastly, the papers collectively provide the researcher, analyst, and designer with a wealth of information and data. The papers are not, nor should they be expected to be, inclusive in any one area. However, they do constitute an integrated source of test methods and procedures for setting design allowables for a variety of composites, with vantage point, Fall 1986.

C. C. Chamis
Aerospace and Composite Structures,
NASA Lewis Research Center, Cleveland, OH 44135; symposium chairman and editor.

Contents

Extreme/Hostile Environment Testing

John F. Mandell,[1] *Dodd H. Grande,*[1] *and Kathryn A. Dannemann*[1]

High-Temperature Testing of Glass/Ceramic Matrix Composites

REFERENCE: Mandell, J. F., Grande, D. H., and Dannemann, K. A., "**High-Temperature Testing of Glass/Ceramic Matrix Composites,**" *Test Methods for Design Allowables for Fibrous Composites: 2nd Volume, ASTM STP 1003,* C. C. Chamis, Ed., American Society for Testing and Materials, 1989, pp. 3-15.

ABSTRACT: Recent advances in ceramic and other high-temperature composites have created a need for test methods that can be used at 1000°C and above. Present test methods usually require adhesively bonded tabs that cannot be used at high temperatures. This paper discusses some of the difficulties with high-temperature test development and describes several promising test methods. Stress-strain data are given for Nicalon ceramic fiber reinforced glass and glass-ceramic matrix composites tested in air at temperatures up to 1000°C.

KEY WORDS: composite materials, test methods, ceramic composites, high temperatures

Fiber composites with brittle matrices, including glass, ceramic, and carbon, have potential properties that may make them attractive structural materials for very high-temperature applications. Recent progress in materials and processing research and development has advanced several systems to a stage appropriate for detailed structural evaluation. Composites used in this study were fabricated in a typical process with unidirectional tapes stacked in the desired ply configuration and compression molded at a temperature where the glass matrix would flow sufficiently to form fully densified material [*1*]. Most fibers can be degraded to some extent at very high processing temperatures, and an effective technique for developing high-temperature resistance is to use glass matrices that can be compression molded at relatively low temperatures, followed by a crystallization step to form a glass-ceramic matrix. A variety of glass and glass-ceramic matrices have been successfully fabricated with several carbon and ceramic fibers [*1-3*].

The mechanical properties of these materials have been evaluated in most studies by three- and four-point flexural specimens, which are convenient to run on small samples of material and at elevated temperatures. While a number of programs are underway to develop high-temperature tests, few studies have reported high-temperature properties other than flexure [*4-7*]. Flexural tests of sufficient span to depth ratio usually produce a tension dominated failure mode with qualitative strength and damage trends that agree with tensile data. However, as with other fiber composites, flexural tests do not give adequate material property data for quantitative structural characterization [*4-8*]. Figure 1 shows the significant difference in flexural and tensile stress-strain curves for specimens taken from a single plate of one of the materials used in this study (the strain in both tests was determined with strain gages). This particular batch of material showed much less than optimum properties as discussed later, but failure was tension dominated in both tests, and both tests would show an increase in ultimate strength of a

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factor of two or more for a more optimized material. The only property that is quantitatively similar in flexure and tension in Fig. 1 is the initial modulus. A full range of tensile, compressive, and shear properties are required to meaningfully evaluate composites for high-temperature applications.

Progress in Test Method Development

A number of factors combine to cause difficulties with high-temperature testing of continuous fiber reinforced composites, particularly those with brittle glass and ceramic matrices:

1. Flexural test data are much less useful than with monolithic glasses and ceramics.
2. Load introduction in unidirectional tension and compression tests is difficult because of the low transverse and shear properties associated with poor fiber/matrix bonding and brittle, high modulus matrices.
3. The unidirectional strength anisotropy is similar to that with polymer matrix composites despite the much higher matrix modulus. As a result, dumbbell-shaped longitudinal specimens have shoulder-splitting problems similar to those with polymer composites.
4. As with standard polymer composites tests, adhesively bonded tabs function well for load introduction. However, bonded tabs require tough adhesives, which are not available with high-temperature properties.
5. Most material systems of interest are currently available only in small plates of 10 to 15 cm length and width at best, which limits load transfer lengths.

The consequence of these problems is that the tensile and compressive stress-strain behavior of longitudinal specimens cannot be evaluated at high-temperatures using established test methods, except for initial modulus up to the matrix cracking strain. Several studies have been conducted [*4,5*] or are in progress [*6–8*] to develop suitable high-temperature mechanical tests. While differing materials systems have been used in these studies, the experiences reported are

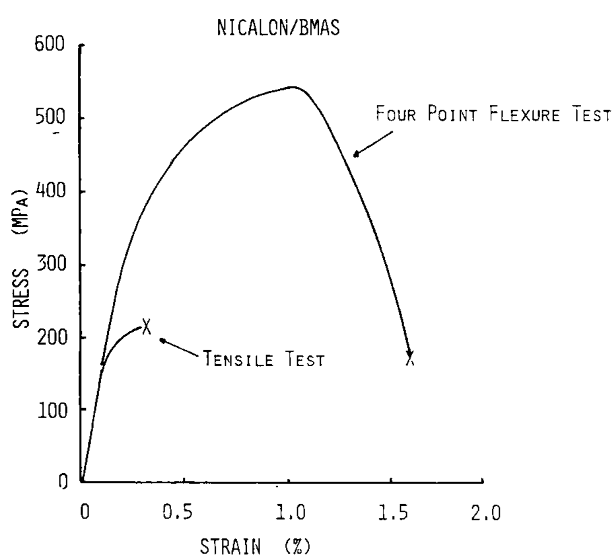

FIG. 1—*Comparison of tensile and flexural stress-strain curves for longitudinal Nicalon/BMAS composite at 20°C.*

generally consistent. The most challenging test appears to be longitudinal tension, where attempts to use dumbbell-shaped specimens have resulted in severely reduced gage section width and thickness, often in several stages with difficult machining and often without achieving consistent gage-section failures [*4–8*]. As discussed later, tension testing with multidirectional laminates using dumbbell specimens has been much less problematical [*4,5,8*].

Simulated shear tests using ±45° or unidirectional off-axis specimens have also been successful [*4*], and it appears that interlaminar shear tests based on short beam [*5*] and double-notched [*5,8*] specimens function properly at low temperatures and could be used at high temperatures. Compressive tests based on unconstrained short dumbbell-shaped specimens appeared to fail in the gage section and give meaningful results, as reported in Ref *4*.

Instrumentation for high-temperature testing is also a potential problem area, particularly at temperatures significantly above 1000°C. Furnaces with air, inert gas, and vacuum conditions, which are compatible with various mechanical testing machines, are commercially available up to at least 2000°C. However, the construction of gripping apparatus may be difficult much above 1000°C unless ceramics can be used, as is commonly done in the flexural test apparatus. Strain measurement creates problems, as strain gages are not available for temperatures in this range. Optical methods are being considered [*8*], but the usual method is extensometers, which are external to the furnace, contacting the specimen with quartz or ceramic rods [*4,6*]. While successful tension tests have been reported up to temperatures of 1100°C [*4*], it should be recognized that these tests usually require long heat-up and cool-down times, so that a typical test takes several hours to conduct.

Materials and Apparatus

Table 1 describes the Nicalon ceramic fibers and 1723 glass and BMAS glass-ceramic matrices used in this study. The composites contained continuous fibers in unidirectional layers with a fiber volume fraction of 0.35 to 0.40 and very little or no porosity. The fibers were poorly distributed in many cases, as described in Ref *9*. These and similar materials have been shown to develop a carbon layer at the interface during processing [*2,3*]. The carbon layer allows the fibers to debond when matrix cracks form, preventing penetration of the matrix cracks through the fibers, which would result in brittle behavior and low strength [*3*].

TABLE 1—*Typical fiber and bulk matrix 20°C properties.*[a]

Parameters	Nicalon® Fiber	1723 Glass Matrix	BMAS III Glass-Ceramic Matrix
Fiber diameter, μm	10 to 15	. . .	. . .
Density, g/cm³	2.55[b]	2.64	2.77
Coefficient of thermal expansion, 10^{-7}/°C	31[c]	45.8	17 (25 to 300°C)
Young's modulus, GPa	193[b]	86	106
Tensile strength, GPa	2.07[b]	. . .	. . .
Failure strain, %	1.0 to 1.3	. . .	. . .
Poisson's ratio	. . .	0.24	. . .
Apparent maximum use temperature, °C	1100 to 1300[c]	600 to 700	1200 to 1250
Composition	silicon-oxycarbide[d]	aluminosilicate, Corning Code 1723	(BaO, MgO, Al_2O_3, SiO_2)

[a]Most data supplied by K. Chyung, Corning Glass Works, except as noted.
[b]Reference *1*.
[c]Nippon Carbon.
[d]Reference *3*.

Test specimens were cut from plates with a diamond-edged cutoff wheel, and ground to the desired shape with a 300 grit diamond wheel. Grinding was done with a water soluble lubricant at a rate of material removal of approximately 5 to 10 μm/pass. Holes in the grip section of some multidirectional specimens were drilled with a diamond grinding bit, while divots for extensometer contact were made with a No. 1 center drill to a depth of about 0.2 mm, and an included edge angle of 110°.

The test system used in this program is a servohydraulic testing machine fitted with an Instron Model 3117 high-temperature furnace system described in Table 2. The system is designed for testing to 1000°C. The tension grips to be discussed later were adapted from Applied Test Systems Model 4053A wedge couplings. All tests were run in air with a heat-up time of 1 to 2 h and displacement rates that produced failure in several minutes (strain rates on the order of 10^{-4}/s).

Tension Specimens and Testing

Figures 2, 3, and 4 illustrate the tension test method used for unidirectional materials [*6*]. The specimen shape has a constant width but a reduced thickness in the gage section produced by wet grinding in small increments. Figure 3 gives the dimensions that were found to yield satisfactory results for 3- to 5-mm thick unidirectional Nicalon/1723 glass and BMAS; the

TABLE 2—*Dedicated high temperature composites testing facility.*

1. Instron Model 1331 servohydraulic testing machine, 10 000-lb (44-kN) capacity
2. Instron Model 3117 high-temperature furnace system (1000°C maximum temperature, air atmosphere)
   - split furnace with self adaptive microprocessor controller
   - water cooled adapters with alignment rings
   - reverse-stress pullrods
   - quartz rod axial and transverse extensometers

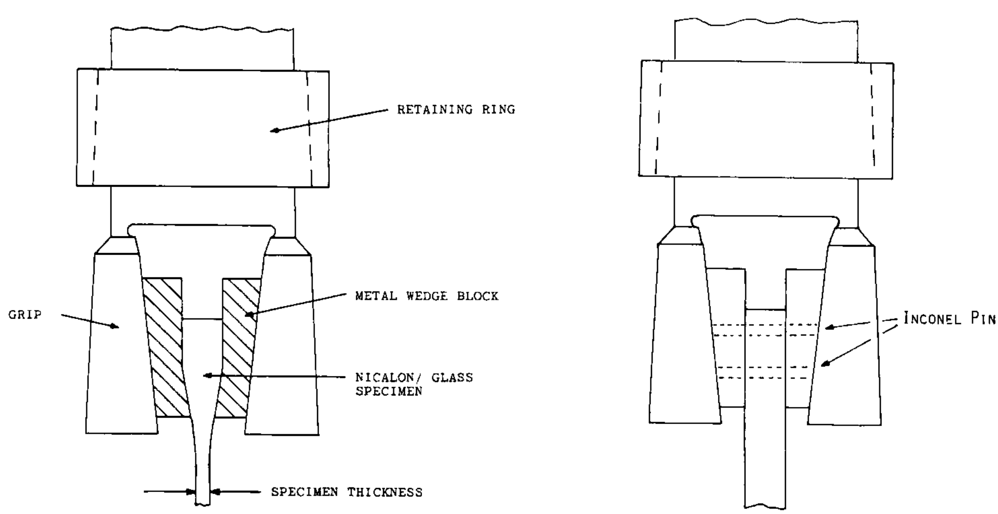

FIG. 2—*Schematics of tension test specimens in grips: thickness-tapered (left) and width-tapered (right).*

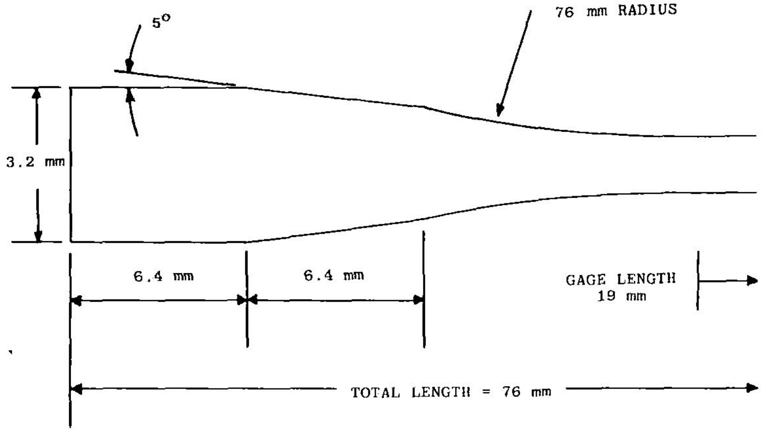

FIG. 3—*Thickness-tapered tension specimen geometry* [6].

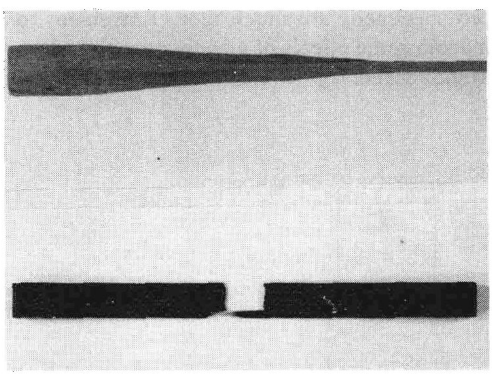

FIG. 4—*Failed longitudinal Nicalon/1723 thickness-tapered specimen: edge view (top) and face view.*

width was a constant 6.4 mm in all cases, and the gage section thickness was 0.75 to 1.3 mm. The thickness is reduced in two stages, first following a 5° taper, then a 76-mm radius with a smooth transition to the gage section. Smooth metal wedge blocks are fit to the straight shoulder and 5° taper sections as shown in Fig. 2*a;* the metal used depends on the test temperature.

When a displacement is applied to the grip, load is introduced through the 5° taper section, which is also compressed normal to the surface to suppress splitting at the shoulder. The 76-mm transition outside the wedge blocks ensures that failure will not initiate from the stress concentration at the edge of the wedge block. The 76-mm radius has proven sufficient in most cases to prevent shoulder splitting in this region for the very slight thickness reduction between the wedge blocks and the gage section. The resulting overall specimen length of 76 mm for a 19-mm gage length is short enough for most small specimens available in this study. A larger transition radius, such as 150 mm, is recommended if larger specimens are available. Most longitudinal and transverse specimens failed in the gage section as shown in Fig. 4, despite some local shoulder splitting evident in this specimen of Nicalon/1723. Many failures were close to the end of the machined radius and some spread into the grips after initiating in the gage section [*6*].

The specimen in Fig. 3 has also been used for 0/90 ply configurations but with an increased tendency toward shoulder failures. With multidirectional laminates it is necessary to provide sacrificial plies to be ground away in the gage section, leaving a balanced laminate configuration. For the case tested, a Nicalon/BMAS plate was fabricated with 16 plies in the configuration $[(90/0)_4]_s$. The average ply thickness was 0.325 mm, but the plies have a slight waviness, so that it was difficult to leave a perfectly symmetrical laminate in the gage section. The actual cases as ground were approximately $[(90/0)_2]_s$ with half-thickness 90° on the outside and $[0/90/0]_s$.

Because of the difficulty and inconvenience of grinding the multidirectional specimens to the geometry in Fig. 3, a second type of tension specimen was also used following Ref *4*. As shown in Figs. 2, 5, and 6, this specimen is ground to a dumbbell shape in the width direction only, with shoulder radii of 13 or 25 mm. The 13-mm radius specimen produced a mixture of gage section failures and failures close to the shoulder; typical failures are shown in Fig. 6. The larger radius specimen appears to fail more reliably in the gage section but with a similar strength; more testing is required to fully investigate the best geometry. Comparison of the width-tapered with the thickness-tapered specimens for Nicalon/BMAS 0/90 material showed a 21% lower strength for the latter, based on the failure stress normalized to the actual amount of remaining 0° material. The lower strength of the thickness-tapered specimens is attributable to difficulties in grinding to produce the desired gage-section laminate configuration caused by wavy ply interfaces. The width-tapered specimens are much more convenient to machine and provide as-molded surfaces, which avoid some effects of grinding problems as well as associated interface oxidation at exposed ground surfaces [*9*]. A width-tapered specimen has been shown to work

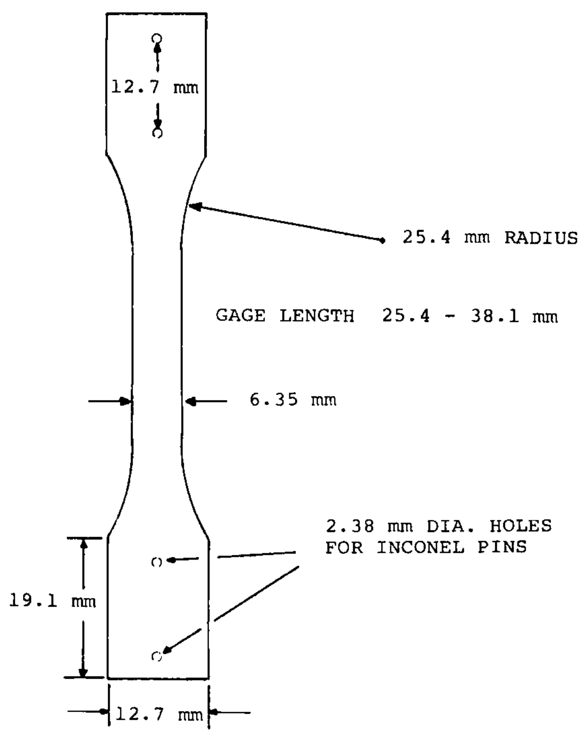

FIG. 5—*Width-tapered specimen geometry (shoulder radius 13 or 25 mm).*

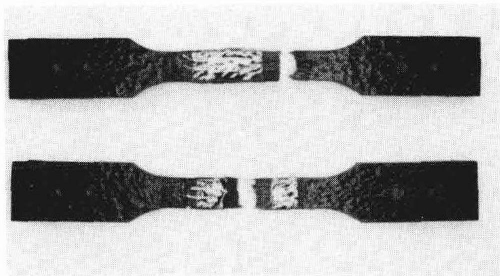

FIG. 6—*Failed 0/90 Nicalon/1723 glass width-tapered specimens.*

successfully in Ref *4* for other ply configurations in addition to the 0/90 and $\pm 45°$ tests run in the present study.

The width-tapered specimen in Fig. 5 was tested in grips shown in Fig. 2*b* with straight, flat metal inserts containing pins. The pins were used to align and seat the specimen, and to hold it during heat-up. The pin arrangement used here was chosen because of the narrow width; three pins were staggered across a wider grip section in Ref *4*. Under load, the wedges tighten and transfer forces through friction rather than through the pins, so that no significant damage was observed at the pins in failed specimens (Fig. 6).

One area of difficulty in conducting tension tests has been with the extensometers. Figure 7 shows the axial extensometer system with detail of the quartz rod contact at the specimen surface. The extensometer rods must be held against the specimen surface divots during thermal expansion as the test system is heated-up for about 2 h, and then must follow the specimen translation as well as the strain between the rods during testing. Initial extensometer alignment and seating must be precise. The extensometer slipped completely out of the surface divots in about 20% of the tests; in some other tests there appeared to be slight movement of the rod tips in the divots, leading to a nonlinear response. Figure 8 shows a typical case of nonlinear extensometer response as compared to strain gage output on the same specimen (at room temperature). Despite these problems, in most cases the longitudinal extensometer appeared to behave satisfactorily over the 20 to 1000°C test temperature range.

Considerably more difficulty was encountered with the transverse extensometer, which is designed for use with round specimen cross sections. When used with the flat specimens to determine Poisson's ratio, the response was not reliable, apparently dominated in many cases by slight twisting or misalignment relative to the specimen edges. This problem may be solved in ongoing efforts to modify the tip geometry of the transverse extensometer quartz rods. In principle, the general approach to transverse strain measurement with this extensometer system should be successful with specimens of this type, but it has not yet received sufficient attention to overcome the current difficulties. The Poisson's ratio data in Table 3 were obtained at room temperature with strain gages.

There are several additional restrictions in using the longitudinal extensometer. The quartz rod tips are brittle and can be chipped or broken if they are in contact with the specimen at fracture. In many tests the extensometer was removed before specimen fracture, with the remainder of the stress-strain curve extrapolated based on the load-stroke curve, which was recorded simultaneously (extrapolated data are shown as a dashed line). Room temperature tests also included front and back strain gages in many cases, and these occasionally showed bending effects in the specimens either from misalignment or nonuniform fiber content through the thickness. The one-sided extensometer cannot detect these effects, so it is desirable to precheck the specimen at room temperature with strain gages at low loads before high-temperature test-