Ceramics International **23** (1997) 283–289
Published by Elsevier Science Limited
Printed in Great Britain
PII: S0272-8842(96)00007-7
0272-8842/97 $17.00+0.00

Property Goals and Test Methods for High Temperature Ceramic Fibre Reinforcement

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(Received 2 August 1995; accepted 27 September 1995)

Abstract: As reinforcement of high temperature composites, ceramic fibres should display high creep strength and rupture strength under potential composite service conditions. Minimum goal values for these properties are developed based on reliable and successful composite application. Conventional and improved test methods for evaluating individual fibres against these goals are discussed and illustrated with recent test results for high performance SiC fibres. Published by Elsevier Science Limited

1 INTRODUCTION

The key to the successful application of high temperature composites (HTC) is the judicious selection and incorporation of ceramic fibre reinforcement with the proper chemical, physical and mechanical properties. The nature and qualitative goals for these properties have been discussed in the literature both for metal (ductile) and ceramic (brittle) matrix composites.[1,2] As in the case for low temperature composites, the most critical fibre property needs are high as-produced strength, high stiffness (modulus) and reliable retention of these properties throughout the service life of the application. Over the last two decades, these needs have led to the development and commercialization of a variety of continuous-length ceramic fibres with both small and large diameters.[2–4] Prime emphasis has been placed on SiC-based and alumina-based fibre compositions in order to achieve high modulus and low density, but more importantly to allow reliable use in oxidizing environments. However, due to their varied manufacturing approaches, these fibres are being produced with many different surface morphologies and internal microstructures, particularly regarding size and content of pores, second phases, grains and grain-boundary phases. Thus, although displaying sufficiently high room-temperature strength and modulus, these fibres show widely different strength retention and stiffness retention (creep resistance) behaviour when exposed to the service conditions envisioned for potential HTC applications.[2,5]

The objective of this article is to shed some light on current and future methods that can be used to evaluate ceramic fibre reinforcement in terms of their ability to display high strength and creep resistance during HTC service. This will be accomplished first by developing some minimum rupture and creep strength goals that individual fibres should meet for reliable and successful application of HTC. Conventional tensile test methods for evaluating fibres against these property goals will then be presented and illustrated with recent test results for SiC fibres of high technical interest. Finally, improved test approaches will be discussed which attempt to minimize deficiencies with conventional methods.

2 STRENGTH AND CREEP RESISTANCE GOALS

At the present time, a convenient approach to the development of quantitative goals for fibre strength retention and creep resistance is through the HTC property requirements for rupture strength and creep strength. These properties, which are dependent on time, temperature and environment, are generally the first properties examined by a design engineer to determine whether a material will be able to perform reliably in a high temperature application requiring long-time structural service. They are typically measured by subjecting the material to a constant stress at a

constant temperature under controlled environmental conditions of technical interest and then observing the times to reach a critical creep strain (creep failure) and the time for the material to finally fracture (rupture failure). From these data, upper-limit stresses (or strengths) can be determined which should not be exceeded if the material is not to creep or rupture fail under the service conditions of a particular application. Generally, for a given service temperature, the lower value between the creep and rupture strength will limit the maximum stress that can be applied on the structural material.

Today there is a lack of analytical constituent theories which relate HTC strength requirements to fibre rupture and creep strength goals. This is the case because fibres within HTC can be subject to time/temperature-varying internal stresses and environmental conditions.[6,7] Until these effects can be adequately modelled, one approach is to use rule-of-mixtures (ROM) composite theory (i.e. fibre–matrix load transfer under isostrain conditions) and to make some simple assumptions concerning matrix behaviour. For example, σ_f, the average stress on the reinforcing fibres within an HTC, can be given by ROM theory as

$$\sigma_f = \sigma_{HTC}/V_f(1 + X) \tag{1}$$

where σ_{HTC} is the uniaxial stress applied on the composite, V_f is the volume fraction of fibres aligned in the stress direction and X is the load share factor, which for elastic behaviour is given by

$$X = E_m(1 - V_f)/E_f V_f. \tag{2}$$

Here E_f and E_m are, respectively, the modulus of the fibre and the effective modulus in the stress direction of the remaining load-bearing composite constituents (primarily matrix). Typically during HTC service, the load share factor will change with time. This can be modelled as changes in the constituent moduli, that is, E_f will decrease slowly in time because of creep; whereas E_m will decrease much more rapidly due to such effects as matrix cracking, plasticity and/or creep. Thus X will also decrease rapidly, leaving the fibres as effectively the only load-bearing constituent. However, V_f is fixed during HTC fabrication, typically ranging from 20% (two-dimensional fibre architecture) to 50% (unidirectional).

It follows from the above discussion that based on reliability considerations, a minimum goal for fibre rupture strength can be developed by assuming that early during HTC service, the composite constituents other than the fibres in the stress direction will lose their load-bearing capability (i.e. $E_m \approx 0$). From eqn (2) this would imply $X \approx 0$, so that by eqn (1),

$$S_f(L) \geq S_{HTC}/V_f \tag{3}$$

where $S_f(L)$ is the average rupture strength of individual fibres measured at gauge length L and S_{HTC} is the rupture strength requirement for the composite. In effect, eqn (3) is the same as the ROM prediction for HTC ultimate strength where the matrix carries no load, but there is still fibre–matrix load transfer. Curtin[8] has shown that although fibre–matrix interfacial conditions and fibre fracture statistics must be taken into account in any accurate composite fracture theory, the ROM prediction is still a good approximation for the ultimate strength of both ductile and brittle composites when the fibre contribution is taken as the average strength of individual fibres taken from the composites and measured near 25 mm gauge length. It follows then that the fibre rupture strength, S_f, in eqn (3) should also be measured near $L = 25$ mm. For a fibre creep strength goal, one can make the same assumptions as above and arrive at the following requirement:

$$C_f(\varepsilon^*) \geq C_{HTC}(\varepsilon^*)/V_f \tag{4}$$

where $C_f(\varepsilon^*)$ is the average creep strength (or stress) for the fibres to reach a critical creep strain, ε^*, under HTC service conditions and $C_{HTC}(\varepsilon^*)$ is the HTC creep strength requirement for the same critical strain limit. Creep limits of 1% or less are often quoted for HTC applications of high technical interest.

Although eqns (3) and (4) can be used to determine quantitative strength goals for individual fibres, it is impossible to generalize their values not only because the HTC strength requirements vary widely from application to application, but also because these requirements are typically only known by the design engineer. However, some generalization can be achieved when one considers the fact that for an advanced HTC to be even considered for a particular application, the design engineer must determine that it will out-perform the best currently available high temperature structural materials, such as nickel-based superalloys. This is the case because in the near term, these conventional materials will be lower cost and afford less performance and maintenance risks than HTC. It follows then that for any potential application, HTC strength properties should be better than those of superalloys. This in turn allows one to conclude that for successful application of

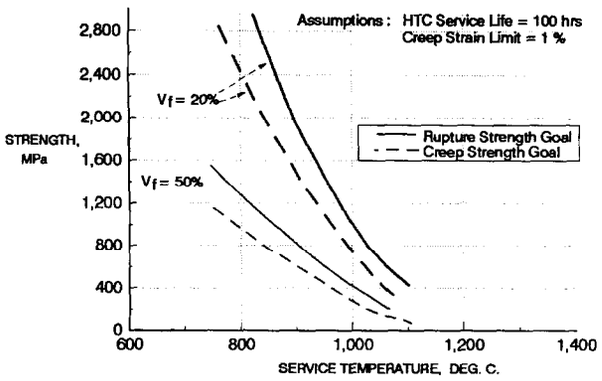

Fig. 1. Minimum rupture and creep strength goals for potential HTC fibres based on scaling from superalloy properties.

HTC, the individual fibres should at a minimum display the following average strength properties:

$$S_f(25\ mm) \geq S_{sa}(t, T)/V_f \tag{5}$$

and

$$C_f(\varepsilon^*) \geq C_{sa}(\varepsilon^*, t, T)/V_f. \tag{6}$$

Here S_{sa} and C_{sa} refer to "superalloy" rupture and creep strengths, respectively, for the service life, t, and service temperature, T, of a particular application.

To illustrate how eqns (5) and (6) may be used to determine fibre strength requirements, one can examine Fig. 1 in which the temperature-dependent solid and dashed curves display the minimum 100 h rupture and creep strength goals for potential HTC fibres. These curves were calculated using (a) eqns (5) and (6), (b) the highest literature values for the temperature-dependent 100 h rupture and creep strengths of cast superalloys,[9] and (c) assumed fibre volume fractions of 20 and 50%. For creep strength, a 1% critical creep strain limit has also been assumed. Thus the average creep strength and rupture strength of individual fibres should exceed the lowest appropriate curve if HTC reinforced by these fibres are to have technical viability. For example, a 2-D HTC with 20% fibre aligned along the principal stress direction would not be competitive with superalloys below 900°C unless the fibre reinforcement displays an average 100 h rupture strength of over 2000 MPa. Obviously, if the HTC application needs only a unidirectional architecture (e.g. $V_f = 50\%$), the fibre strength requirements are less stringent.

3 CONVENTIONAL CREEP AND RUPTURE TEST METHODS

For evaluation of the creep and rupture strength of individual fibres, the conventional test procedure is

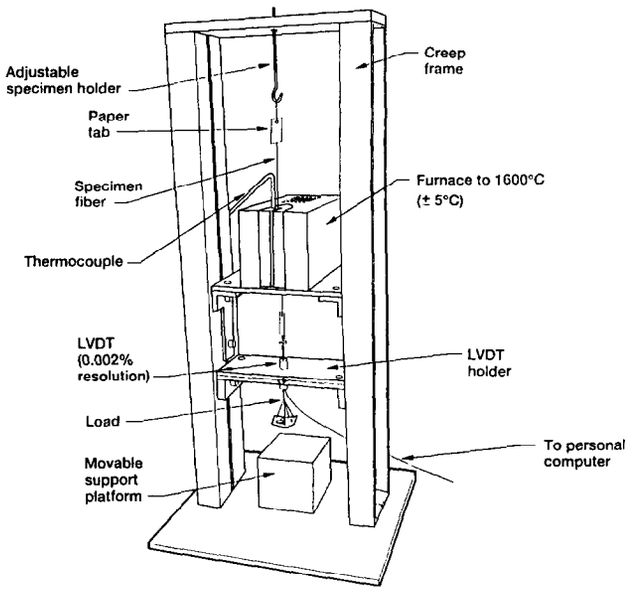

Fig. 2. Single fibre creep/rupture rig using dead-weight loading.

to subject an individual fibre specimen of length L and diameter D to a constant tensile load P at a constant test-temperature T and to measure fibre elongation u versus time t until the fibre finally fractures at rupture time t_R. Creep strain is then determined from

$$\varepsilon_c = u(t, T, \sigma, G)/L \tag{7}$$

where $\sigma = 4P/\pi D^2$ is the applied stress and G symbolizes effects from the environmental gases. Rupture time typically is also a function of temperature, stress, length and environment, i.e.

$$t_R = t_R(T, \sigma, L, G). \tag{8}$$

Rupture time displays these dependencies because of the statistical nature of flaw size and distribution along the length of the as-produced fibre and because these flaws can increase in size with time, temperature, stress and environment.[10] For fibre creep strength, data described by eqn (7) can then be used to yield

$$C_f(\varepsilon^*) = \sigma_c(\varepsilon_c = \varepsilon^*, t_c, T, G) \tag{9}$$

whereas for fibre rupture strength, eqn (8) data will yield

$$S_f(25\ \text{mm}) = \sigma_R(t_R, T, L = 25\ \text{mm}, G). \tag{10}$$

Here σ_c and σ_R are the average fibre stresses, respectively, to reach ε^* in service time t_c and to cause rupture in service time t_R. The specimen length at the test temperature has been chosen to be ~ 25 mm.

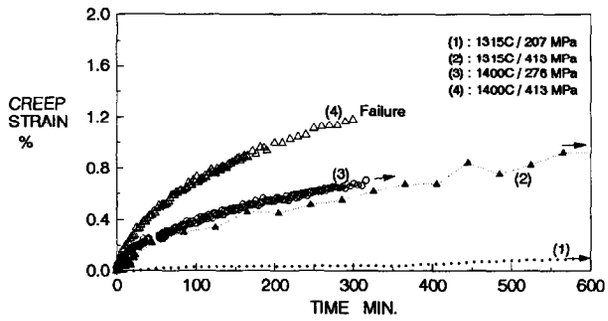

Fig. 3. Typical creep curves for Hi-Nicalon SiC fibres.

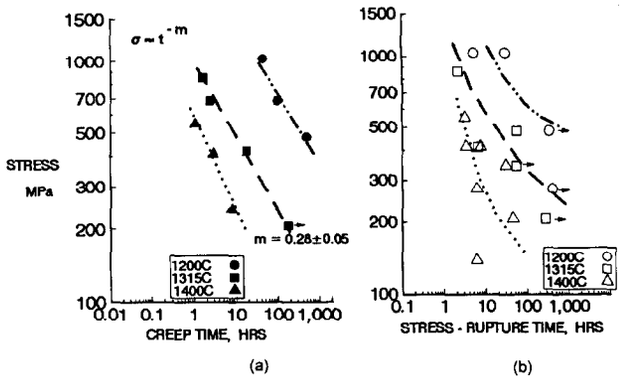

Fig. 4. 1% creep strength (a) and rupture strength (b) for Hi-Nicalon SiC fibres.

Regarding the physical aspects of a conventional creep-rupture test, the simplest method of applying the tensile load is by dead-weight loading of a vertically-held fibre. A typical dead-weight test set-up for air testing[11] is shown in Fig. 2. High temperature contact between the fine diameter fibre and other materials is generally avoided because of the possibility of extraneous interaction effects. Thus in Fig. 2, "cold" grips are used in which each end of a long fibre (> 100 mm) is glued to paper tabs that are then attached to simple hooks. The fibre is placed in an air-capable furnace (e.g. $MoSi_2$ heating elements) which can provide a constant and nearly uniform temperature region along the centre section of the fibre (e.g. hot zone of ~25 mm). Fibre temperature is measured and controlled at the centre of the hot zone, typically with a thermocouple placed very close to the fibre. Elongation and fracture of the fibre can be measured at the bottom paper grip or at the dead-weight by a variety of non-contact high-precision electronic displacement gauges, such as LVDTs and devices which utilize laser or optical sensing. Care is taken to avoid any displacement of the upper grip during the test duration. Also, use of cold grips requires a correction to the measured creep data in order to account for creep displacements that occur along the entire fibre length.[12]

After the fibre specimen is positioned at room temperature under near-zero loading, the conventional test procedure is (a) to heat the fibre to the test temperature, (b) then, after a short soak time, to load the fibre to full load, and (c) to monitor fibre elongation until fracture or until a convenient test duration time is reached (typically ~100 h). If possible, it is desirable to save the tested and/or fractured section of the fibre. This will allow, for example, the use of advanced microscopy techniques to determine the effects of the test conditions on fibre diameter and surface morphology, and perhaps provide insight into the underlying creep and rupture mechanisms. Obviously the equipment in Fig. 2 can also be set up for testing in environments other than air to study the effects of other gases on fibre behaviour. Long-term vacuum and/or inert (argon) testing will generally require the use of graphite or refractory metal furnace elements. For any environment, it is important that it be fully characterized and be maintained stable during the test.

Typical creep strain and 1% creep strength data measured in air using the above test methods are shown in Figs 3 and 4(a), respectively, for the commercially available "Hi-Nicalon" SiC-based fibre.[13] This small diameter (14 μm) fibre, which is produced by Nippon Carbon in multifilament tow (~500 end count), is currently being examined as a potential reinforcement for HTC with ceramic matrices. The noise in the Fig. 3 data is caused primarily by specimen vibration due to air convection currents (chimney effect). Although this effect produces some small data scatter, the Fig. 4(a) creep strength data shows that much greater statistical scatter (sometimes as high as ±100%) can often be seen from fibre to fibre in the same production run. This can most likely be attributed to microstructural variations along the fibre length and/or across the many fibres of a multifilament tow. For the stress versus rupture time data, even more scatter is observed, as shown by the Hi-Nicalon results in Fig. 4(b). Such scatter implies the need for multiple tests for each fibre type in order to better understand the distribution in creep/rupture behaviour and to develop more accurate creep/rupture models.

Using the test results of Figs 4(a) and (b), one can now evaluate the Hi-Nicalon fibre against the superalloy-derived creep and rupture strength goals given by eqns (5) and (6). This is shown in Fig. 5 where the fibres are evaluated for two-dimensional HTC (effective V_f of 20%) which must operate for 100 h in air with a 1% critical creep limit. From this figure, it can be concluded that whether the application is creep or rupture limited, the Hi-Nicalon fibre from ~1000 to 1400°C should

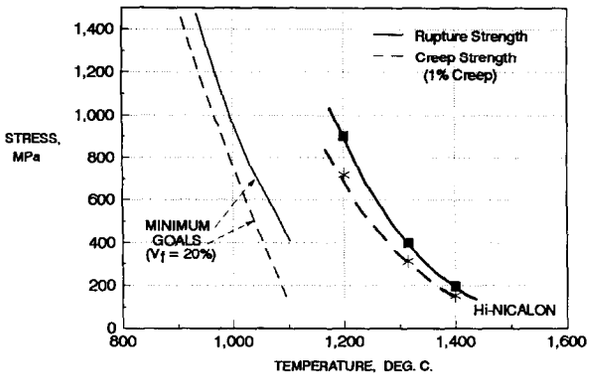

Fig. 5. 100 h creep (1%) strength and rupture strength of Hi-Nicalon SiC fibres compared against minimum goals for HTC with $V_f = 20\%$.

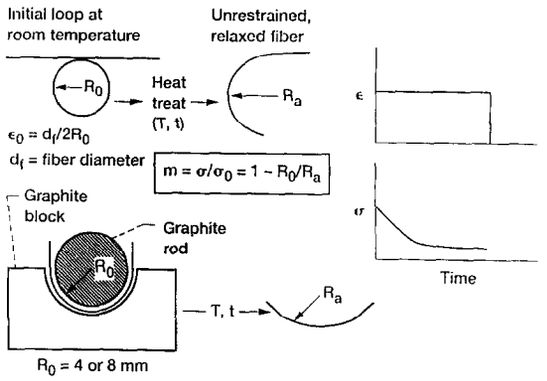

Fig. 6. Bend stress relaxation (BSR) test for reinforcement fibres.

be able to provide HTC that can structurally outperform superalloys. However, this does not imply that the Hi-Nicalon HTC will have sufficient performance for any application requiring operation above ~1000°C. In this regard, plots such as those in Fig. 5 should be generated for a variety of fibre types and potential HTC service conditions so that the fibre type with the best combination of strength properties for a particular application could be selected.

Another important factor to be considered is that Hi-Nicalon data were generated for as-produced fibres which were not exposed to the HTC fabrication or internal service conditions. Since for some fibre types, these conditions can significantly alter fibre structural performance, it is important that the Fig. 5 plots also be generated for fibres which were pre-treated under simulated HTC fabrication conditions and then tested under simulated HTC service conditions.

Finally, from Fig. 5 it can be concluded that for the assumed application conditions, the structural performance of the Hi-Nicalon fibre is limited by creep and not by rupture. Indeed, for creep limits less than 1%, the difference in Hi-Nicalon creep and rupture strengths would be even greater.

4 IMPROVED TEST APPROACHES

In order to better understand and predict fibre thermostructural behaviour within HTC, new methods are currently being implemented for creep/rupture testing. These include approaches related to the fibre itself, such as (a) fibre pre-treatment for times and temperatures which simulate HTC fabrication conditions, (b) fibre testing under gaseous environments which simulate HTC external and internal conditions, and (c) the testing of fibres and multifilament tows that are, repectively, thinly-coated or infiltrated with matrix material in order to simulate HTC interface and matrix effects. Also being examined are variations in the test procedures including (a) controlled changes in stress and temperature conditions to simulate in-situ HTC thermomechanical conditions and to develop approaches for modelling these changes, (b) laser speckle extensometry to remotely measure fibre creep strain in a ~100 μm section of the hot zone,[14] and (c) bend stress relaxation (BSR) testing[15] which, as described below, allows very convenient and simple evaluation of fibre creep performance.

A schematic of the BSR test is shown in Fig. 6. For this test, short lengths of fibre (2–5 cm) are held at constant surface strain in a pure bending mode while being thermally treated for a specific time at constant temperature in a controlled environment. For small diameter fibres, bending modes of different applied strains can be achieved by tying the fibres into small loops of different radii R_o. For large diameter fibres, graphite or alumina jigs can be used to impose bend strains. After thermal exposure, a creep-induced radius-of-curvature, R_a, is observed in the fibre at room temperature. For a time t at test temperature T, it is convenient to define a BSR ratio m given by

$$m(\varepsilon_o, t, T) = 1 - R_o/R_a = \sigma(t, T)/\sigma(0, T). \quad (11)$$

Here $\varepsilon_o = D/2R_o$ is the applied bend strain on the surface of a fibre of diameter D, $\sigma(0,T)$ is the average elastic bend stress initially in the fibre and $\sigma(t,T)$ is the average elastic stress remaining in the fibre after the long-time thermal exposure. Thus the BSR ratio m is a measure of stress relaxation in the fibres, ranging in value from unity for elastic behaviour ($R_a = \infty$) to zero for significant creep behaviour ($R_a = R_o$).

For assessment of fibre creep behaviour, the simple BSR test has many advantages. First, in comparison to conventional tensile creep tests, it

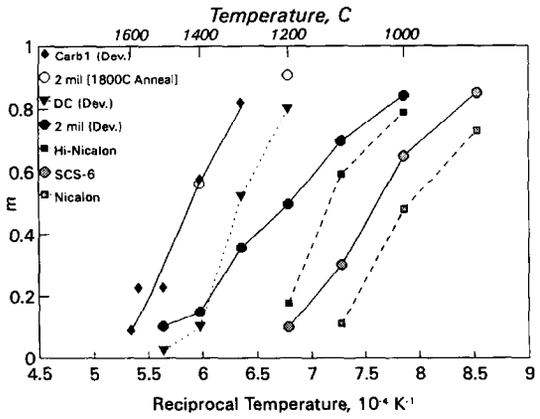

Fig. 7. Comparison of commercial and developmental SiC fibres based on 100 h BSR *m*-ratio versus temperature.

eliminates the need for furnaces with long uniform hot zones, for mechanical grips, for remote sensors and for multiple experimental runs that are often required to establish time, temperature and stress dependencies and also to determine statistical variations. These advantages can be especially important for newly developed fibres which are often not very strong, nor available in large quantities or in long lengths. Second, for polycrystalline fibres, which generally creep with stress power dependencies near unity, the BSR *m*-ratios are effectively independent of applied strain and thus equal to those stress relaxation ratios that would be measured in a pure tensile test. Finally, because a large portion of a fibre's stress relaxation occurs in the primary creep stage, the time–temperature dependencies of the BSR ratio can be used at creep strains below 1% to monitor microstructural changes and to rank polycrystalline fibres according to their creep strengths.

For example, because of the test simplicity, BSR data have been obtained for essentially all ceramic fibres of high technical interest.[16,17] These data have typically been measured as a function of temperature and applied bend surface strain for exposure times from 1 to 100 h. To appreciate how the various fibres generally differ in creep behaviour, one can examine Fig. 7 which compares 100 h BSR data for some commercial and developmental SiC-based polycrystalline fibres.[16]

These data, which were found to be independent of the test environment (argon or air), show that the SiC-based fibres can vary significantly in creep-limited temperature capability. Although there is much variation in average grain size for these fibres, it would appear that the major differences in BSR (and creep) are related to low-viscosity second phases, such as oxygen-containing complexes in the polymer-derived and air-cured Nicalon fibre; free silicon and carbon in the chemically vapour-deposited SCS-6 fibre; and perhaps free carbon in the polymer-derived Hi-Nicalon and the developmental oxygen-free Dow Corning (DC) fibres. Thus the fibres with the highest creep resistance and creep strength are those with stoichiometric SiC composition and no grain boundary second phases, such as the developmental Carborundum (Carb) sintered α-SiC fibre and the Textron (2-mil) CVD fibre after pre-treatment at 1800°C to allow carbon to leave the grain boundaries.[18]

5 CONCLUSIONS

Although this article presents minimum strength goals and test methods for evaluating ceramic fibres for high temperature composites, more research efforts are needed to determine whether these approaches are sufficient to quantitatively, or even qualitatively, judge the structural performance of the total composite system. This is the case because in real applications of the total composite system, the fibres will not be subjected to the simple test conditions described here, such as constant stress, temperature and environment. Nevertheless, it would appear that these simple approaches can be used at least for an initial evaluation and ranking of potential fibre behaviour. Final evaluation will come only after composites are fabricated with the highest ranked fibre types and then tested under the service conditions of a selected application.

ACKNOWLEDGEMENTS

The author would like to acknowledge the invaluable professional contributions of H.M. Yun and G.N. Morscher in the preparation of this article.

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