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Short-Time Creep Behavior of Carbon, Graphite, and Silica Phenolic Composites at Elevated Temperatures

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Laboratory Operations
THE AEROSPACE CORPORATION

Prepared for SPACE AND MISSILE SYSTEMS ORGANIZATION
AIR FORCE SYSTEMS COMMAND
LOS ANGELES AIR FORCE STATION
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FOREWORD

This report is published by The Aerospace Corporation, El Segundo, California, under Air Force Contract No. F04701-69-C-0066.

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Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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Titan III System Deputy Program Director
Deputy for Launch Vehicles

ABSTRACT

Short-time tensile creep behavior of carbon, graphite, and silica phenolics at elevated temperatures was determined for durations up to 40 sec. A plasma arc was used as a heat source, with test temperatures ranging from 1095 to 2760°C. The results show appreciable amounts of creep strain, especially for the higher temperature and stress levels. The magnitude of these strains suggests that a complete structural analysis should take account of creep and creep rupture.

A surprising result is that the activation energies of creep for carbon, graphite, and silica phenolics are experimentally identical. Observation of identical creep activation energies for composites based on these widely different fibers is contrary to intuitive expectations, since the fiber is usually thought to be dominant in mechanical behavior.

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I. INTRODUCTION

The results of tests of short-time tensile creep in the warp direction of carbon, graphite, and silica phenolics at elevated temperatures are reported. To the authors' knowledge, there are no available elevated temperature creep data on the phenolic materials used for reentry nosetips and rocket engine nozzle liners. A plasma arc was used to heat the test specimens in time periods typical of the rapid heating experienced by components fabricated from such materials during atmospheric reentry or rocket engine firing. Creep strains as large as 9% were observed for time periods up to 40 sec. The strains, as well as the creep rates, were found to increase with increasing temperature and stress for all materials tested. Activation energies, determined from the creep rates, were about 33 Kcal/mole for all three materials.

Data for the graphite phenolic show that the activation energy for creep is independent of the applied stress. Further, for graphite and silica phenolics, the stress dependence of the creep rate is governed by a power law in which the creep rate varies as the third power of the stress.

II. EXPERIMENTAL PROCEDURE

The test apparatus employed in the creep tests is the same as that for the tensile stress-strain tests described in [1]. Briefly, a high-temperature plasma arc was used to heat the central section of a test specimen with a temperature rise time typical of the rapid heating during reentry and rocket engine firing. Tensile load was provided by a pneumatically actuated loading frame, and the surface temperature of the specimen central gauge section was monitored with an automatic recording pyrometer. Loading was initiated only after the central specimen temperature had stabilized. The rise time of the load step was typically 1.5 sec. The test duration, up to 40 sec, was limited by overheating of the associated test fixtures. The strain history was obtained from motion pictures of the specimen test section taken for the duration of each test. Protrusions on the specimen sides with drilled holes established the effective gauge length. Typical temperature, loading, and strain (thermal expansion and creep) histories are shown in Figure 1.

Pertinent data on the phenolic composites used in this study are shown in Table 1. All of these composites are pressure-cured woven cloth preimpregnated with the phenolic matrix materials. The carbon phenolic specimens were machined from a 12- (warp direction) × 10- (fill direction) × 4-in. (perpendicular-to-plies direction) billet fabricated by Haveg Industries, Inc. The graphite specimens were machined from a large graphite phenolic rocket motor throat ring set aside for test purposes because of an interlaminar crack on the inside of the ring. The silica specimens were machined from a portion of an Aerojet-General Corp. (AGC) ablative skirt for a rocket nozzle. Typical specimen gauge dimensions were 0.40 in. long (warp direction), 0.20 in. wide (fill direction), and 0.15 in. thick (perpendicular-to-plies direction).

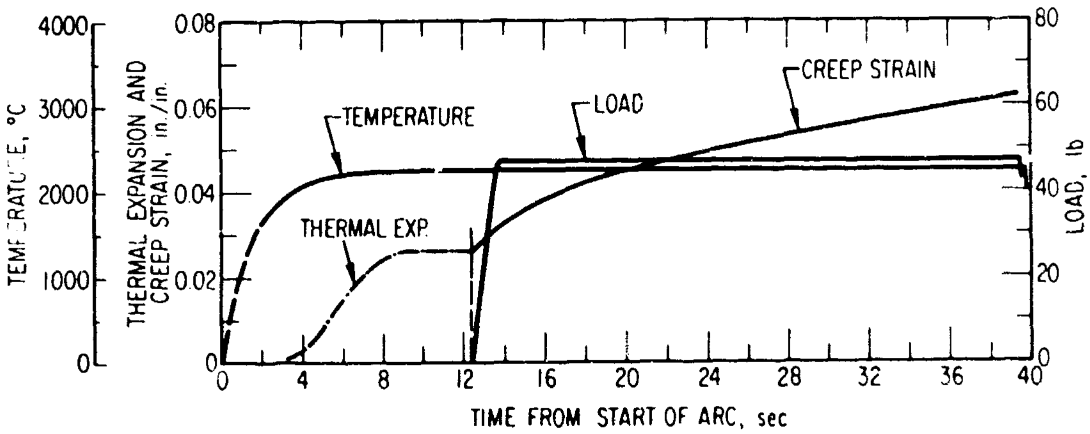

Figure 1. Typical Temperature, Loading, and Strain Histories (Carbon Phenolic Data)

Table 1. Phenolic Composites Used in Creep Tests

Material	Fabricator	Cloth	Resin	Post-Cure Temp.
1. R-6300 carbon	Haveg Industries	3M Co. square-weave carbon	Monsanto SC-1008	350°F
2. FM 5014 graphite	Hitco	Union Carbide square-weave graphite	Ironside 91-LD	275°F
3. silica	Haveg-Reinhold	Sil-Temp square-weave silica	Monsanto SC-1008	320°F

III. RESULTS

Figure 2 shows, for temperatures of 2270 and 2760°C, the tensile creep behavior of carbon phenolic for a stress about 55% of the ultimate tensile strength of the material at temperature. The creep curves for graphite phenolic, shown in Figure 3, were obtained for temperatures ranging from 2080 to 2710°C and stress levels from 45 to 75% of the ultimate tensile strength at temperature. Figure 4 shows the creep curves for silica phenolic, which were obtained at 1095 and 1310°C for stress levels of 45 to 70% of the ultimate tensile strength at temperature.

The creep curves of Figures 2 - 4 show approximate linearity over the last 15 sec. The creep rates $\dot{\varepsilon}$ were determined from these final linear slopes of the creep curves. Plots of $\dot{\varepsilon}$ versus 1/T for the three materials are shown in Figure 5. Activation energies obtained from the slopes of the lines in this plot are shown in the figure. From the limited present data, these energy values, ranging from 27 to 35 Kcal/mole, could be considered experimentally identical, even though these phenolic composites are fabricated from widely different fibers. This is surprising, as the fibers are usually thought to be dominant in mechanical behavior. The plots of the graphite phenolic data in Figure 5 are nearly parallel, showing that the apparent activation energy is independent of the applied stress.

The stress dependence of the creep rate at given temperatures for graphite and silica phenolics is shown in Figure 6. Figures 5 and 6 imply that the creep rate during the latter stages of the test may be described by the following equation:

$$\dot{\varepsilon} = K_0 \, \sigma^3 \exp(-\Delta H/RT) \qquad (1)$$

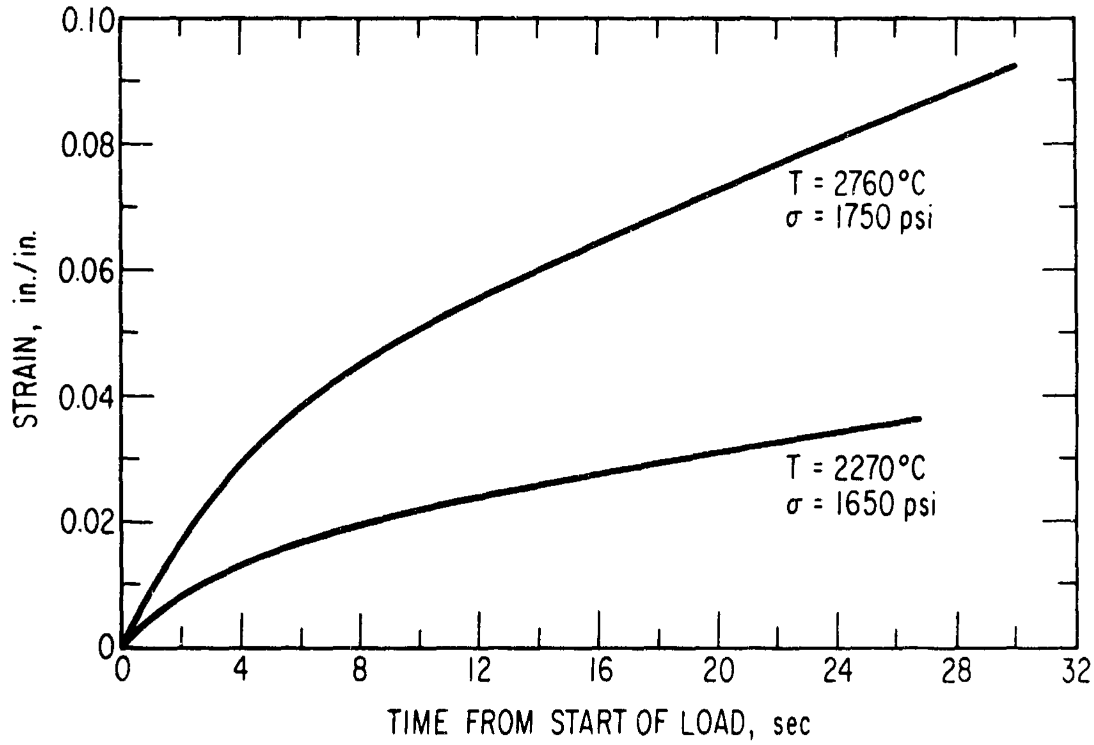

Figure 2. Tensile Creep Behavior of R-6300 Carbon Phenolic in the Warp Direction at about 55 Percent of Ultimate Tensile Stress at Temperature

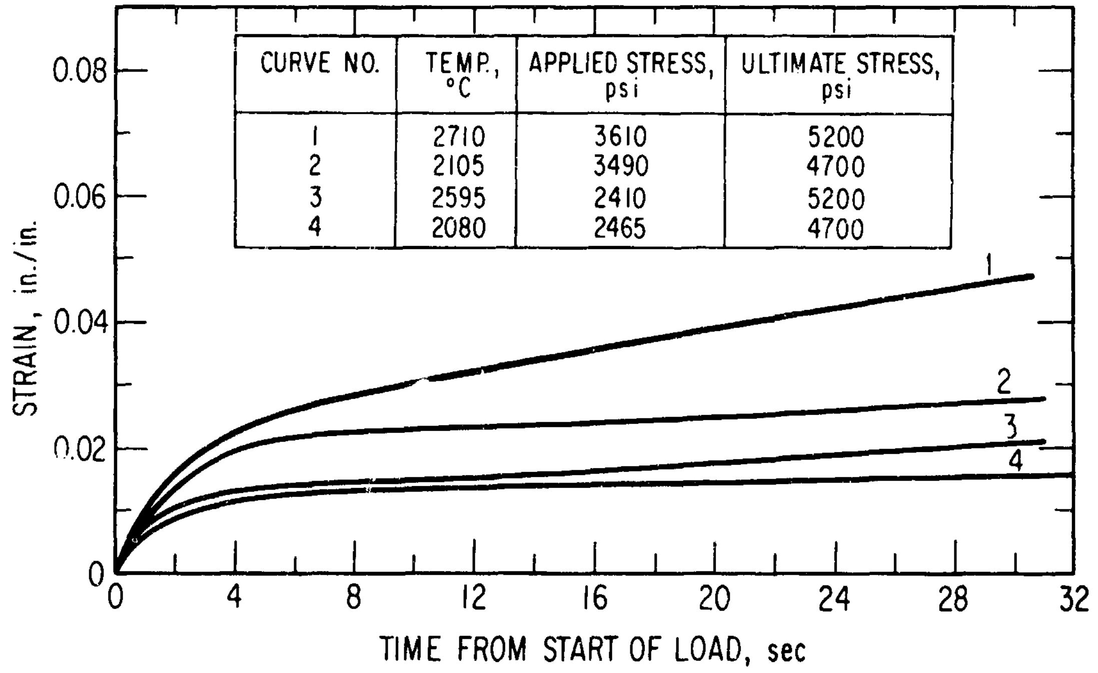

Figure 3. Tensile Creep Behavior of FM 5014 Graphite Phenolic in the Warp Direction

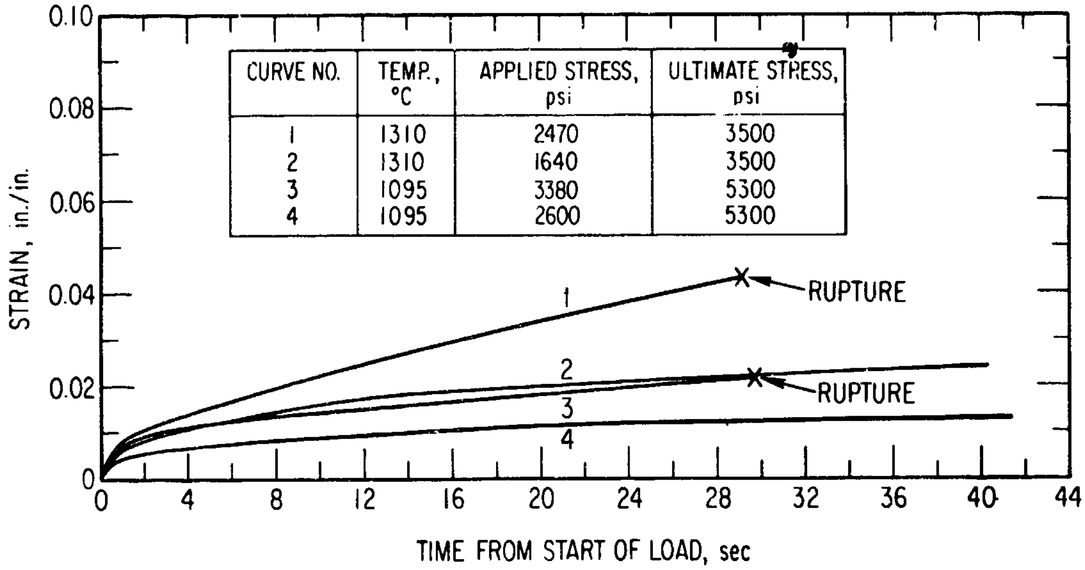

Figure 4. Tensile Creep Behavior of AGC Silica Phenolic in the Warp Direction

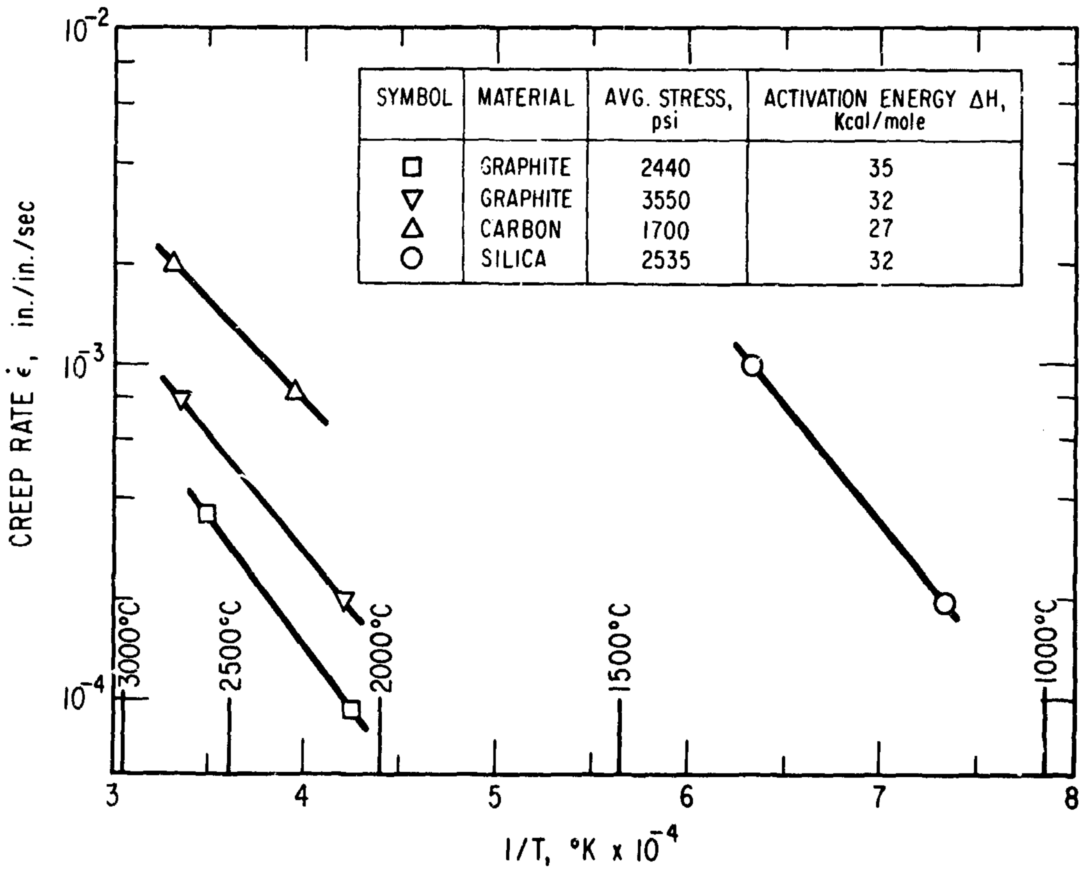

Figure 5. Plot of Creep Rate $\dot{\epsilon}$ vs 1/T for Carbon, Graphite, and Silica Phenolics

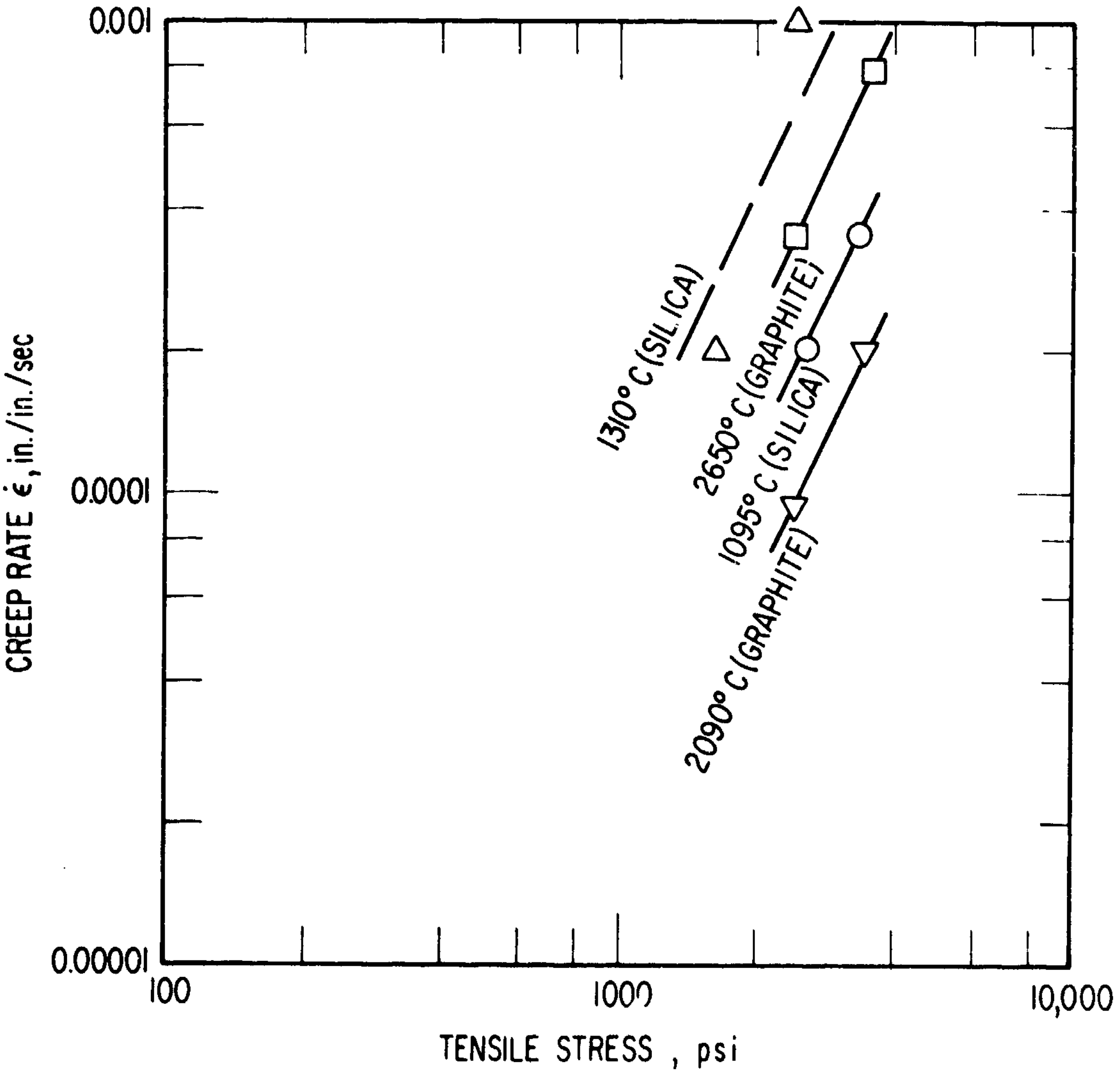

Figure 6. Creep Rate $\dot{\epsilon}$ vs Tensile Stress for Graphite and Silica Phenolics at Various Temperatures

where

$K_0 = 9.38 \times 10^{-14}$, in./in./sec/(psi)^3

σ = stress, psi

R = ideal gas constant = 1.99 cal/mole/°K

T = temperature, °K

$\Delta H = 33 \times 10^3$ cal/mole

A few creep runs were conducted in the compressive mode. The general characteristics were similar to results obtained in the tension mode, such as amount of creep, but not enough runs were conducted to verify the numerical consistency of creep parameters such as K_0 and ΔH. These experiments imply, however, that the results are not simply caused by the experimental procedure, nor by swelling effects at temperature.

IV. DISCUSSION AND CONCLUSIONS

While the data are somewhat limited in extent and should be interpreted as tentative, it is clear that these phenolic matrix materials will exhibit substantial creep at temperatures and stresses of practical interest. This factor is significant in design. Further, the observed phenomena are of fundamental interest. Activation energies for tensile creep of various graphites are known to range from 124 to 240 Kcal/mole [2, 3]. If, in the present experiments, the creep of the fiber were the rate-controlling process, the activation energy for the creep of the composite should be of this same order instead of the observed values of about 33 Kcal/mole. The most likely explanation for experimentally identical activation energies is that, in the char regime, the charred phenolic matrices are identical and are the rate-controlling phases. This is particularly true for the carbon and graphite phenolic composites.

Prediction of creep is essential to efficient design with existing materials, while control of creep, in conjunction with other properties, has significance in synthesis of improved materials. Further study of these phenomena is expected to aid in understanding and synthesis of present and future composite materials.

No simple explanation for the mechanism of the creep behavior, particularly the apparent third order dependence on stress and the low activation energy, seems to be available. The present results suggest that creep of composites is a fruitful area for theoretical and experimental investigations that could yield methods of predicting stress relief (or dimensional instability) produced by creep.

REFERENCES

1. A. Ching and W. E. Welsh, Jr., "Strength and Stress-Strain Properties of Rapidly Heated Laminated Ablative Materials," AIAA Journal, Vol. 5 (1968) p. 2312-2315.

2. E. J. Seldin, "High Temperature Tensile Creep of Graphite," Wright Air Development Div., WADD-TR-61-72, Vol. XVIII (March 1964).

3. H. E. Martens, L. D. Jaffe, and D. D. Button, "High-Temperature Short-Time Creep of Graphite," Jet Propulsion Lab., California Institute of Technology, Pasadena, Calif., Progress Report 20-373 (December 1958).

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13. ABSTRACT

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