THE STRESS DEPENDENCE OF THE TENSILE CREEP RATE IN CARBONS AND GRAPHITES[*]

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Little is known about the deformation mechanisms which are responsible for the high temperature ductility of carbons and graphites. In those materials which show appreciable tensile reduction in area (pyrolytic and vitreous carbons, etc.), the observed elongation must result directly from mechanisms capable of producing extensive homogeneous strain. The stress (σ) dependence of the creep rate ($\dot{\epsilon}$) can be useful in identifying the active deformation mechanisms in such materials. Using the empirical relationship

$$\dot{\epsilon} = A\sigma^n \tag{1}$$

the value of the stress exponent n is characteristic of the deformation mechanism. For example, $n = 1$ is expected for the Nabarro-Herring diffusion mass transport mechanism, while $n \sim 4$ is expected for a dislocation climb mechanism such as is believed to occur in many metals. In materials which deform with negligible reduction in area (conventional and stress-recrystallized coke-pitch graphites, etc.) any true plasticity which occurs may be highly localized and the observed macroscopic strain behavior may differ from that of the rate-controlling microscopic mechanism. In such materials, the interpretation of the stress exponent is more difficult.

A study of the stress dependence of the creep rate is being made on pyrolytic carbon (pyrolytic graphite), stressed parallel to the substrate, and on vitreous (glassy) carbons. Pyrolytic carbon deforms at approximately constant volume to at least 20% elongation; vitreous carbon deforms with about two thirds of the constant volume reduction in area. A simple stress-change technique is used in which changes in $\dot{\epsilon}$ resulting from alternate increases and decreases in σ are observed as a function of temperature, stress range, and thermal and strain history of the specimen. A strain-gage extensometer, coupled to the grips, is used to record instantaneous strain as a function of time. Possible deformation of the grips is minimized by using vitreous carbon inserts at the contact interface between the specimen and the grips. Ink fiducial marks on the specimen are used to determine the actual total uniform gage elongation after each test. In general, two or three separate creep runs, each involving several stress changes, are made on each specimen. A two-stage dead weight loading device is used to add and remove stress increments. A high transient creep rate is often observed during the first 2-3 minutes after a stress increase. A recovery period with an abnormally low (or negative) creep rate follows a stress decrease. These abnormal transient rates are not used in determining the stress dependence. In general, the ratio of the creep rate before to that after a stress change, and the ratio of the two stresses are used to determine the stress exponent. On occasion, absolute rates and stresses are also used. In either case, a plot of creep rate as a function of strain has been found very useful in determining the proper creep rates. Temperatures in the range 2400 - 2900°C are achieved with a graphite tube resistance furnace in inert gas, and read with an optical pyrometer.

The values obtained for n in both pyrolytic and vitreous carbons are lower than the $n = 5-8$ values reported by Green and Zukas for various

coke-pitch graphites which show little or no tensile reduction in area.
Two deformation regions are of interest in pyrolytic carbons stressed in
tension parallel to the substrate. The first stage of deformation (approx-
imately the first 10% elongation) of as-deposited material is characterized
by extensive straightening of the layer planes (dewrinkling) and graphitiza-
tion (layer ordering and crystallite growth). Second stage deformation
(total elongations greater than 10%) occurs in well graphitized, very highly
oriented material. The layer planes are aligned closely parallel to the
stress axis. The stress exponent is difficult to determine during the first
stage because the creep rate is changing rapidly with strain; in the particu-
lar pyrolytic carbon used in this investigation, there is a strain-rate
maximum in this region. In the second deformation stage the creep rate
changes more slowly, decreasing monotonically with strain under constant
load. Conformance with equation (1) was verified at 2600°C over the stress
range 10,000 - 20,000 psi in the second stage region. Most subsequent data
were taken at only two stress levels in each test run, generally in the
ratio of approximately 1.5 : 1. Results obtained to date indicate that
the stress exponent n in pyrolytic carbons lies between 1 and 2. No
clear dependence of n on strain has been found over the elongation range
5 - 20%. However, n does appear to vary systematically with temperature,
decreasing from ~ 2 at 2500 - 2600°C to ~ 1.5 at 2700°C and ~ 1 at 2800°C.
These results suggest that the Nabarro-Herring mechanism contributes signi-
ficantly to the tensile deformation of graphitized pyrolytic carbon parallel
to the layer planes. However, other mechanisms characterized by $n > 1$
also appear to operate under appropriate temperature and stress conditions.
These mechanisms have not yet been identified. The temperature dependence
of the creep rate in the second stage of deformation corresponds to an
activation energy value in the range 240 - 280 kcal/mole. This is in
excellent agreement with values obtained earlier for creep in the first stage
of deformation, and for the graphitization process. Together with the
evidence for Nabarro-Herring creep, this provides further strong support
for a diffusion mass transport mechanism with an activation energy of approx-
imately 260 kcal/mole in graphite. All of the results discussed above were
obtained using as-deposited specimens in which the deformation and associated
microstructural changes are confined to the gage length region. Specimens
machined from pyrolytic carbon which had been annealed at 2950°C for one hour
under a small compressive stress normal to the substrate behave quite
differently. A stress exponent value of about 4 was obtained. About 80% of
the recorded elongation in these samples results from compressive kinking
parallel to the substrate plane in the gripped regions, and it seems likely
that the high n value is associated with this deformation mode. High n
values, then, do not necessarily imply a dislocation climb mechanism.
Preliminary results have also been obtained on the stress dependence of
the creep rate in two vitreous carbons (GC-20 and GC-30) in the 2600 - 2700°C
temperature range. For these carbons, n values in the range 2 - 3 were
found.