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A Study on Friction Coefficient Measurement of Refractory Ceramics at High Temperatures

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고온에서 내화요업재료의 마찰계수 측정에 관한 연구

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초 록

고온에서 내화요업재료의 마찰계수 측정장치를 고안하였다. 이 장치는 고온에서 Hoop stress를 동시에 측정할 수 있는 이점을 갖고 있다. 이 장치로 240°C～1800°C에서 Graphite의 마찰계수를 측정하였다. 마찰계수는 온도가 증가함에 따라 감소한다.

1. INTRODUCTION

In a previous paper[1)] a hoop stress apparatus was described and equations were introduced for the high temperature testing of ring specimens of chemically vapor deposited (C. V. D.) tungsten tubes or refractory ceramics. Standard tensile specimens are very difficult to prepare from such tubes, and many problems are associated with tensile testing of refractory ceramics and metals at high temperatures. Holman and co-workers[2)] have developed an apparatus for room temperature hoop tests of C. V. D. rings, but the mechanical properties of tubular C. V. D. materials at elevated temperture had not been evaluated heretofore. This new apparatus has the additional advantage of being adaptable to measure the friction coefficient values of materials at high temperature. The primary objective of the current work was to determine the effective friction coefficient values of graphite in the temperature range of 140°C to 1800°C.

2. APPARATUS

Figure 1 shows a schematic diagram of the high temperature hoop stress apparatus. A downward force is applied to a tapered graphite cone which transmits force to eight equal segments that press out uniformly on the inner surface of the ring specimen putting the ring into tension. These components are shown in more detail in Figure 2. Figure 3 shows a schematic diagram of the high temperature tensile test apparatus. The transformation from hoop testing to tensile testing requires only replacing the support block and graphite cone by pull rods and specimen grips.

For descriptive purposes the apparatuts is divided into the following parts: furnace chamber and heating system, load train, and temperature measurement.

The furnace chamber is basically a 10 inch diameter stainless steel pipe, 13 inch high with 3/8 inch thick stainless steel flanges. A stainless steel plate is sealed with an O-ring to the top flange of the chamber. Motion of the upper pull (or compression) rod is achieved through a stainless steel bellows. The pull rod is a 15/16 inch diameter stainless steel rod, seven inches long. The bottom flange of the chamber is O-ring sealed to a large stainless steel plate. A 2 inch long, 15/16 inch diameter stainless steel pull rod is welded to the bottom plate as shown in Figure 1. Two flanged openings are provided on opposite sides of the heating chamber. A pyrex window facilitates observation (for temperature measurement with an optical pyrometer) as well as vacuum and/or gas atmosphere introduction. The other flanged opening admits the induction coil. Water cooled copper tubing is wound around the outside and on the top of the furnace chamber.

A tantalum susceptor, heated by induction, radiates heat to the specimen. The power supply is a 20 Kilowatt Lepel Induction generator with a frequency of approximately 360,000 cycles per second. A 4-turn copper tubing coil with an inner diameter of 3 inches and a length of 1 inch was used for tensile specimens. The seamless tantalum susceptor had a 1/8 inch sighting hole to permit temperature measurement by an optical pyrometer.

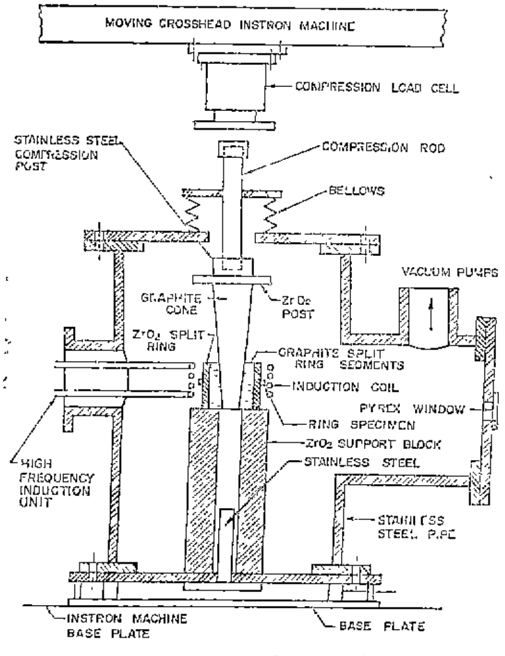

Fig. 1. High-temperature hoop testing apparatus.

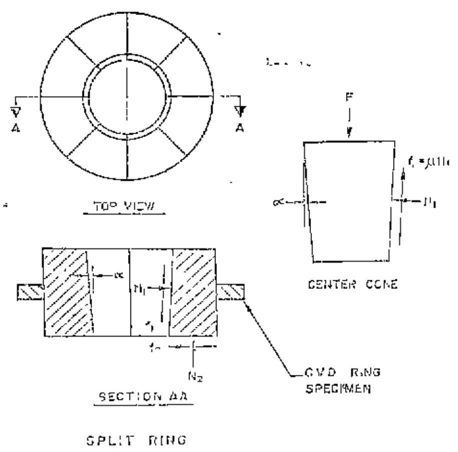

Fig. 2. Depails of sprit graphite ing, graphite cone and ring specimen in the components of hoop testing apparatus.

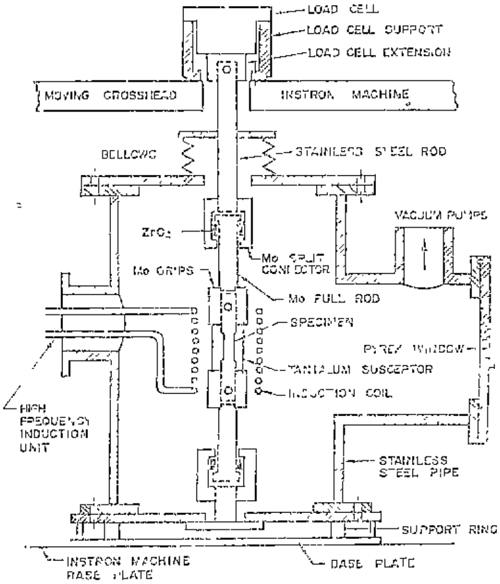

Fig. 3. High thmperature tensile testing apparatus.

The entire hoop stress load train assembly is shown in Figure 1. The top of the stainless steel compression rod is threaded to the main compression rod. A ZrO_2

post, 2 inches in diameter and 1 inch high, is placed between the end of the compression rod and the top of the graphite cone to provide thermal insulation. Eight equal split ring graphite segments of a split ZrO_2 ring are placed between the graphite segments and the C.V.D ring specimen in order to prevent any possible carburization reaction between the graphite and the C.V.D tungsten. A ZrO_2 tube served as a support for the two split rings, the specimen and the cone.

The entire tensile test load train assembly is illustrated in Figure 3. The top and bottom stainless steel pull rods are threaded into molybdenum alloy couplings. The latter are linked to molybdenum alloy pin grips.

C. V. D tungsten tubes of 1. 5 inches inside diameter and 0. 050 inch wall thickness and flat 0. 040 inch thick strips of CVD tungsten were purchased from the San Fernando Laboratories of Fansteel Metallurgical Company.

Ring specimens, 1/4 inch high were cut using a lathe tool post grinder with a 0. 020 inch thick silicon carbide cutoff wheel operating at approximately 7000 RPM. The C. V. D. tungsten tube as held in a soft collet. Gage length and pin holes for flat tensile specimens were spark cut. Any slight damage to the specimen caused by spark cutting (and tool post cutting) was removed by electro-polishing (20 gr NaOH+980cc distilled water). The electopolishing produced a reduction of thickness of approximately 0. 002 inches.

With the load train and specimen installed, the furnace chamber was exacuated, helium gas was admitted to the chamber, the specimen was slowly heated to and then held at the test temperature for a period of five minutes to establish thermal equilibrium. The friction coefficient of measurement and graphitehoop stress were performed with an Instron Tensile Machine at a cross-head speed of 0. 01 in. /min.

3. DERIVATION OF THE HOOP STRESS EQUATION

If a downward force F is appied to the graphite cone, see Figure 2, the conditions for equilibrium require:

$$F=A_1N_1(\sin\alpha+\mu_1\cos\alpha) \qquad \text{Eq. (1)}$$

where A_1 is the area of the tapered surface in contact with the split graphite ring, N_1 is the normal pressure on the tapered graphite surface of the cone, α is the angle of graphite taper and μ_1 is the friction coefficient between the graphite cone and the graphite split ring.

Considering the graphite split ring; the conditions for equilibrium require:

$$A_2N_2=A_1N_1(\sin\alpha+\mu_1\cos\alpha) \qquad \text{Eq. (2)}$$

where A_2 is the total area of the bottom of the split graphite ring and N_2 is the normal pressure on that graphite area. Similarly, equilibrium requires that for the split graphite ring, in the radial direction·

$$A_rP+A_2N_2\mu_2=A_1N_1\cos\alpha-A_1N_1\mu_1\sin\alpha \qquad \text{Eq. (3)}$$

where A_r is the inner area of the ring specimen, P is the pressure on the inside of the ring, and μ_2 is the friction coefficient between the split graphite ring and the support block.

Dividing Equation (3) by Equation (2), using the fact that from Equations (1) and (2) $F=A_2N_2$, and assuming that μ_1 equals μ_2^*, then P is expressed by:

$$P=\frac{F}{A_r}\left[\frac{(1-\mu^2)\cos\alpha-2\mu\sin\alpha}{\sin\alpha+\mu\cos\alpha}\right] \qquad \text{Eq. (4)}$$

If Equation (4) is substituted into the hoop stress equation:

$$\tau_{hoop}=\frac{P\cdot\gamma}{t} \qquad \text{Eq. (5)}$$

where τ is the circumferential stress, γ is the internal radius of the ring specimen, and t is the ring thickness, then the hoop stress is expressed by:

$$\tau_{hoop}=\frac{F[(1-\mu^2)\cos\alpha-2\mu\sin\alpha]}{2\pi ht(\sin\alpha+\mu\cos\alpha)} \qquad \text{Eq. (6)}$$

where h is the height of the ring. Equation (6) thus has two unknowns, τ_{hoop}, and μ, the friction coefficient of graphite. The friction coefficient of graphite can be obtained from following relation, assuming the tensile yield stress is equal to the value of the hoop stress at yielding.

$$\mu=\frac{-(\sigma/k\cos\alpha+2\sin\alpha)+[(2\sin\alpha+\sigma/k\cos\alpha)^2-4\cos\alpha(\sigma/k\sin\alpha-\cos\alpha)]^{1/2}}{2\cos\alpha} \qquad \text{Eq. (7)}$$

where k is F/2ht and σ is the yield stress, separately obtained from a tensile test.

It may be helpful to point out that there is an upper bound on the thickness which appears in Equation 5. Timoshenko[3] showed an equation for the circumferential stress, σ_θ, in an open ended cylinder of inner radius a and outer radius b, subjected to internal pressure

P_1:

$$\sigma_\theta=\frac{a^2P_1}{b^2-a^2}\left(1-\frac{b^2}{r^2}\right) \qquad \text{Eq. (8)}$$

This stress is a maximum on the inner surface; however, if b=1.1a, $\sigma_{\theta\max}>\sigma_{\theta\min}$ by only 10.5%. Thus, there is a small error if we assume σ_θ to be uniformly distributed over the thickness and given by

$$\sigma_\theta=\frac{P_1a}{b-a} \qquad \text{Eq. (9)}$$

which is the same as our hoop stress equation (5). In the rings we have been testing a is approximately 0.75'' and t is approximately 0.050''; therefore, b/a=1.07 which is less than that Timoshneko's recommended upper bound of 1.1 for the safe assumption of a uniform stress distribution.

It might be argued that friction between the segments and the inside of the test ring should produce a frictional stress whose effect would be to lower the yield stress of the ring. We know firstly, that such friction of graphite must be static if in fact it exists, because no relative motion is found between the graphite segments and the graphite ring. Secondly, a consideration of the sign of such a frictional stress in the ring shows that it would be compressive and maximum on the inner surface of the ring Recall that Timoshenko's[3] σ_θ was, in the general case, a maximum on the inner surface and tensile in nature. Thus, even if such a friction of graphite exists at this location, its effect would be to make σ_θ even more uniform than envisioned by Timoshenko for the case of frictionless internal pressure on the interior of a thin walled cylinder.

4. EXPERIMENTAL RESULTS AND DISCUSSION

The relation between friction cefficient and hoop stress was first studied at room temperature for the metals, aluminum, copper, and brass. The tensile and hoop stress specimens were produced from the same plate for each metal. The apparatus components in this preliminary room tempreature prototype were of stainless steel. The results of tensile tests for these metals were self-consistant and reproducible. The average value of 0.2% offset yield stress for brass was 48,000 psi, for aluminum 38,000 psi, and for copper 17,000 psi. These values were substituted into Equation$_{(7)}$ to obtain the friction coefficients for hoop stress measurements at room temperature. The results of the effiective friction coefficient were as follows: for copepr it varied from 0.17 to 0.19, for aluminum from 0.12 to 0.14, and for brass from 0.11 to 0.12. Thus, each metal produced a very consistent and reproducible effective friction coefficient value. We do not think that this implies that the friction at room temperature is affected by the particular materials involved in the segment-metal ring pair, but rather that the effective friction coefficient of the apparatus is force sensitive. At any temperature the major sliding is beween the cone and segments, for the prototype this would be stainless steel versus itself, the friction coefficient, μ, can be sometimes a function of normal force, contrary to our usual concept of this coefficient. For example, it has been shown[4] that the friction coefficient of diamond decreases as the load increases, approximately following a law $\mu=KW^{-1/3}$, where W is the load. In the current case, μ dropped from the range of 0.06—0.19 to that of 0.104—0.12 as the yield stress(indicative of normal force on cone) increased from that of copper (17—18 kpsi) to that of brass (46—50 kpsi). If one uses the upper values from each range of μ and each range of yield stress and calculates the constant in the previous relation, one finds a difference in K values of only 12.7%, which suggests that the stainless steel behavior is similar to diamond in the force sensitivity of its friction coefficient.

Since these results indicated the feasibility of friction coefficient measurement at room temperature, the technique was extended to temperature up to 1800°C.

Figure 4 shows the temperature dependence of the 0.2% offset yield stress of flat specimens. The yield stress data in the temperature range of 1400°C to 1800°C are in good agreement with Taylor's[5] results for powder metallurgical (P.M.) tungsten. Taylor and coworkers had investigated the mechanical properties of P.M. tungsten in the temperature range of 1400°C or above. Also, the yield stress of 9,600 psi at 800°C and 8,500°C psi at 1,000°C are in good agreement with

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*By far, the major relative motions occur between the graphite cone and the split grophite ring. This assumption will be unnecessary in future work where the support block will also be graphite and the ZrO_2 split ring will not be in contact with the latter.

Shim's[6] results for C. V. D. tungsten.

These values of yield stress were substituted into equation(7) to obtain the friction coefficients of graphite at high temperatures. The results of the effective friction coefficient of graphite were as shown in Table 1. If we plotted the values of graphite friction coefficient as function of temperatures, it can be seen that friction coefficient of graphite has temperature dependence and as shown in Fig. 5. Fig. 5 shows the temperature dependence of the friction coefficient of graphite and μ decreases linearly with increasing temperature. The graphite produce a very consistant and reproducible effective friction coefficient values at high temperatures. It is believed that very consistant and reproducible value of friction coefficient value at high temperature is due to the graphite mechanical properties such as high strength by compression force at high temperatures. The experimental results are similar to Kenyon's[7] results for outgased graphite.

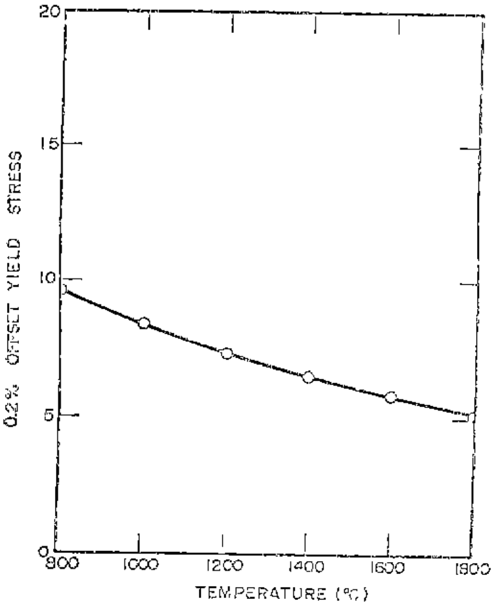

Fig. 4. Temperature dependence of 0.2% yield strength of flat specmens.

Table 1. Friction Coefficient of Graphite

Temp.	α	Temp.	α
230°C	0.45	1200°C	0.19
500°C	0.36	1400°C	0.14
800°C	0.28	1600°C	0.12
1000°C	0.24	1800°C	0.08

CONCLUSIONS

1. The experience gained during the development of this apparatus strongly indicates that the apparatus is extremely well-suited to the mechanical properties determinations on all chemically vapor deposited or refractory ceramic materials.
2. This apparatus may be well suited to future application in the determination of friction coefficients of high temperature materials.
3. The yield stress can be readily calculated from hoop stress tests.

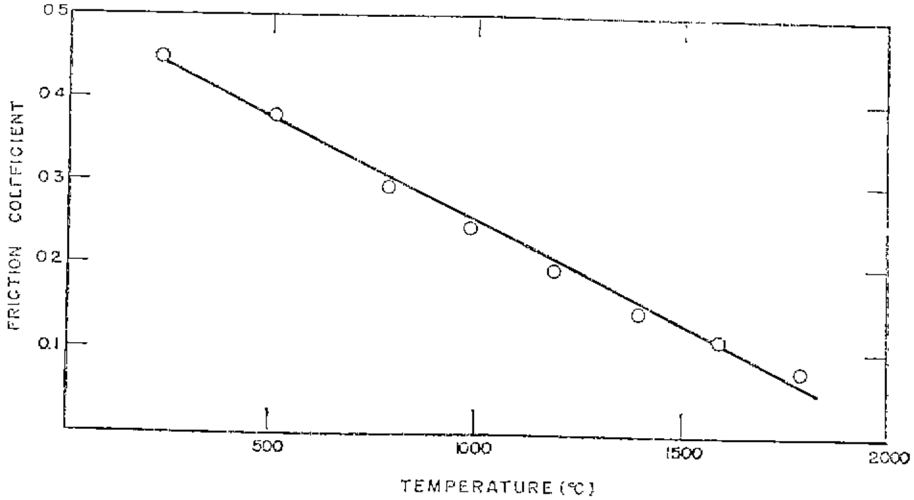

Fig. 5. Temperature dependence of the friction coefficient of graphite.

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