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RESEARCH MEMORANDUM

CORRELATION OF PHYSICAL PROPERTIES OF CERAMIC MATERIALS WITH RESISTANCE TO FRACTURE BY THERMAL SHOCK

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CORRELATION OF PHYSICAL PROPERTIES OF CERAMIC MATERIALS WITH RESISTANCE TO FRACTURE BY THERMAL SHOCK

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SUMMARY

An analysis is made to determine which properties of materials affect their resistance to fracture by thermal stresses. From this analysis, a parameter is evaluated that is correlated with the resistance of ceramic materials to fracture by thermal shock as experimentally determined. This parameter may be used to predict qualitatively the resistance of a material to fracture by thermal shock.

Resistance to fracture by thermal shock is shown to be dependent upon the following material properties: thermal conductivity, tensile strength, thermal expansion, and ductility modulus. For qualitative prediction of resistance of materials to fracture by thermal shock, the parameter may be expressed as the product of thermal conductivity and tensile strength divided by the product of linear coefficient of thermal expansion and ductility modulus of the specimen.

INTRODUCTION

The operating temperatures of aircraft propulsion systems are limited by the melting temperature of the alloys currently available. In general, higher operating temperatures result in higher cycle efficiencies for gas turbines. One means of obtaining these higher efficiencies is to select materials that are more refractory than the alloys currently used. Oxides and carbides of metals are therefore being considered for use in aircraft propulsion systems because of their high melting temperatures.

In the selection of a ceramic for use as a turbine-blade material, the properties of the material that affect its operating characteristics must be considered. Because sudden engine starts and stops are relatively frequent during service operation, the resistance of blade materials to fracture by thermal shock is of considerable importance. The stresses induced in a material by the thermal shocks of unsteady engine operation are the result of heat flow from the center of the blade to the cooler surrounding atmosphere upon stopping, or from the hot gas to the blade upon starting. This time-dependent type of heat flow is commonly termed "unsteady-state heat flow," and the stresses induced are different from those that would be induced by having steady flow from a constant-temperature hot zone to a constant-temperature cold zone (steady-state heat flow).

The methods of evaluating resistance to thermal shock have not as yet been standardized. A general method of determining thermal-shock resistance of a material consists in subjecting a specimen to a number of cycles of alternate heating and cooling until fracture occurs. (The variations in the procedure are considerable.)

Other investigators have presented work showing the dependence of material properties on resistance to thermal shock. In reference 1, a derivation of the theory is presented for the tendency of bricks to spall. It is shown that the spalling tendency is proportional to the coefficient of thermal expansion divided by the product of the maximum tensile strain and the square root of the diffusivity.

According to reference 1, spalling is usually defined as a fracture of a refractory brick or block resulting from any of the following causes: (1) temperature gradients in the brick, (2) compression in a structure of refractory bricks sufficient to cause shear failures, and (3) variation in coefficient of thermal expansion between surface layer and body of the brick. The A.S.T.M. method of classifying brick consists in expressing resistance to spalling as weight loss after a specified spalling test. This weight loss is the result of flaking of the surface layer, or chipping of the corners of the brick.

Fracture instead of spalling has been observed in investigations dealing with the thin ceramic specimens that are of interest as gas-turbine-blade materials. For this reason, resistance of thin ceramic specimens to fracture by thermal shock is of greater interest than their resistance to spalling.

An analysis was therefore made at the NACA Lewis laboratory to determine which properties of a ceramic material affect the resistance to fracture by thermal stresses. The stresses caused by heat flow are considered in the analysis reported.

SYMBOLS

The following symbols are used in this analysis:

A	area, (sq in.)
c_p	specific heat at constant pressure, (Btu/(lb)($^{\circ}$F))
d	size factor
E	ductility modulus of specimen, (lb/sq in.)
E_A	ductility modulus of element A, (lb/sq in.)
E_B	ductility modulus of element B, (lb/sq in.)
F	force, (lb)
F_A	force in element A, (lb)
F_B	force in element B, (lb)
h	diffusivity, (sq ft/hr)
k	thermal conductivity, (Btu/(hr)(sq ft)($^{\circ}$F/in.))
l	length, (in.)
q	convection heat-transfer coefficient, (Btu/(hr)(sq ft)($^{\circ}$F))
S	stress in specimen, (lb/sq in.)
S_A	stress in element A, (lb/sq in.)
S_B	stress in element B, (lb/sq in.)
s	tensile strength of specimen, (lb/sq in.)
T	temperature of specimen, ($^{\circ}$F)
T_A	temperature of element A, ($^{\circ}$F)
T_B	temperature of element B, ($^{\circ}$F)
T_0	temperature of gas surrounding specimen, ($^{\circ}$F)
t	time, (sec)

u	velocity, (ft/sec)
x	direction of heat flow
y, z	directions normal to direction of heat flow
α	linear coefficient of thermal expansion, (in./in./°F)
ϵ	emissivity of body
μ	viscosity of gas
ρ	density, (lb/cu in.)
σ	Stefan-Boltzmann constant, 0.173×10^{-8} (Btu/(sq ft)(hr)($^{\circ}R^4$))

ANALYSIS

The thermal stresses induced during a thermal-shock test are the result of temperature gradients from the center of the specimen to the surface, and not of temperature variations along the surface. An examination of the equations for heat flow for specific boundary conditions yields information about the dependence of heat flow on some independent measurable material properties.

Material Properties Affecting Thermal Shock

For the unsteady-state condition, the equation for heat flow in a small element of the material is

$$\frac{\partial T}{\partial t} = h\left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2}\right) \qquad (1)$$

where, for the body,

$$h = \left(\frac{k}{\rho c_p}\right)\left(\text{dimensional constant}\right)$$

For a thin specimen such as the disk used in this investigation, the heat flow is essentially linear and equation (1) becomes

$$\frac{\partial T}{\partial t} = h\,\frac{\partial^2 T}{\partial x^2} \qquad (2)$$

For the case where the outer surface is cooled suddenly, reference 2 shows that the temperature at any point in the body is a homogeneous function of h and t. Moreover, $\frac{\partial T}{\partial x}$ and $\frac{\partial^2 T}{\partial x^2}$ are homogeneous functions of the same variables. This result indicates that the diffusivity does not influence the value of the maximum gradient in the body, but only the time at which it occurs.

The stresses that are induced in a material by the temperature gradients are considered in determining the strength requirements for adequate resistance to fracture by thermal shock. It is assumed that two adjacent elements A and B of a material are subjected to unequal temperatures, the two elements are at temperatures T_A and T_B (higher than T_A), respectively (fig. 1), and all properties of A and B are independent of temperature. If elements A and B were free to expand, the expansion of B would be greater than that of A as a result of the higher temperature of B. If these elements are restrained by each other, the expansions of the elements A and B must be the same to a first approximation. The solution of the problem of determining the thermal stresses in two elements of a body, as shown in figure 1, is based on two conditions: (1) the tensile force in element A equals the compressive force in element B; and (2) the total elongation of A equals that of B.

In order to satisfy condition (1),

$$F_A = F_B \tag{3}$$

The thermal expansion of element A is

$$\alpha l(T_A - T_0)$$

The thermal expansion of element B is

$$\alpha l(T_B - T_0)$$

The extension of element A caused by the tensile force exerted on it is

$$\frac{F_A l}{A\ E_A}$$

where

$$\frac{F_A}{A} = S_A$$

The compression of element B caused by the compressive force exerted on it is

$$\frac{F_B l}{A\ E_B}$$

where

$$\frac{F_B}{A} = S_B$$

In order to satisfy condition (2),

$$\alpha l (T_A - T_0) + \frac{S_A l}{E_A} = \alpha l (T_B - T_0) - \frac{S_B l}{E_B}$$

or (4)

$$\alpha(T_A - T_0) + \frac{S_A}{E_A} + \frac{S_B}{E_B} = \alpha(T_A - T_0) + \alpha\Delta T$$

where

$$\Delta T = T_B - T_A$$

Then

$$\frac{S_A}{E_A} + \frac{S_B}{E_B} = \alpha\Delta T$$

Because elements A and B comprise a free body in static equilibrium,

$$S_A = S_B$$

Then

$$2\ \frac{S_A}{E} = \alpha\Delta T$$

For a unit distance,

$$S_A = \frac{\alpha E}{2}\ \frac{\Delta T}{\Delta x}$$

In the limiting case of infinitesimal distance,

$$S_A = S_B = S = \frac{\alpha E \frac{dT}{dx}}{2} \qquad (5)$$

For a given value of S, the resistance to cracking by thermal shock is determined by the tensile strength of the material. In order for S to be low, $\alpha E \frac{dT}{dx}$ must be low.

Fluid Properties Affecting Thermal Shock

The usual equation for heat conduction at the surface in one dimension is

$$-k \frac{\partial T}{\partial x} + q(T - T_0) + \sigma \epsilon (T^4 - T_0^4) = 0 \qquad (6)$$

where

$q(T - T_0)$ convection heat loss

$\sigma \epsilon (T^4 - T_0^4)$ radiation heat loss

An exact solution for a problem in heat conduction in the unsteady state with accurate radiation boundary conditions has not yet been found (reference 3). The analysis presented herein considers only the shock encountered in cooling. Inasmuch as radiation is disregarded, the results are semiquantitative only.

For the one-dimensional case, the convection heat loss $q(T - T_0)$ is equal to $\frac{k \partial T}{\partial x}$ and the temperature gradient normal to the length of the specimen is equal to

$$\frac{\partial T}{\partial x} = \frac{q}{k} (T - T_0) \qquad (7)$$

The convection heat-transfer coefficient q is usually expressed in terms of the following gas properties:

$$q = f(u, d, \rho, \mu, k, c_p)$$

For a given size and shape of specimen at a given gas velocity and specific heat, the convection heat-transfer coefficient may be considered independent of temperature (reference 4).

From equation (7) for a low temperature gradient $\frac{\partial T}{\partial x}$ at the surface, a low convection heat-transfer coefficient and a high thermal conductivity are desirable properties of the materials.

From this reasoning, for maximum resistance to cracking by thermal stresses, the desirable properties of a material are high tensile strength, low coefficient of thermal expansion, low modulus of elasticity at failure, high thermal conductivity, and low convection heat-transfer coefficient.

The ratio of stress to strain at failure is designated the ductility modulus and can be evaluated when the stress and the strain at failure are known, as shown in figure 2.

From the analysis presented, a material parameter that includes the thermal conductivity, tensile strength, thermal expansion, and the ductility modulus of the materials can be used to predict qualitatively the susceptibility of a material to cracking by thermal shock. This parameter can be expressed as $\frac{ks}{\alpha E}$.

APPARATUS AND PROCEDURE

The equipment used to determine the thermal-shock resistance of ceramic materials is shown in figure 3. This unit consists of a furnace for heating the specimen to the evaluation temperature and an air-quenching system for rapid cooling of the specimen. A specimen holder made from a high-temperature alloy is used to support the specimen and to transport it from the furnace to the air-quenching stream and back. The specimens used for evaluation in this unit were disks 2 inches in diameter and 1/4 inch thick.

The procedure used in the thermal-shock evaluation (reference 5) consisted in subjecting the specimen to a number of cycles of alternate heating and quenching until failure occurred. The specimen was introduced into the preheated furnace where it was held at the evaluation temperature for 10 minutes, after which it was transported to the quenching air stream and held for 5 minutes. Preliminary tests indicated that in this procedure the center of a specimen was heated from room temperature to 1800° F in approximately 6 minutes and was cooled from 1800° to 400° F in $1\frac{1}{2}$ minutes and from 400° to 85° F in $2\frac{1}{2}$ minutes. Each thermal-shock cycle was followed immediately by the next cycle at the same temperature. The appearance of a crack constituted failure of the specimen. When a specimen had withstood 25 thermal-shock cycles of a given evaluation temperature, the test was continued at an evaluation temperature 200° F higher for 25 cycles or until failure, whichever occurred first; if no failure occurred, this evaluation procedure was continued at the next higher temperature.

The thermal-shock specimens were inspected visually after each quenching. When there was doubt as to the presence of a crack, the specimen was examined by means of radiography.

RESULTS AND DISCUSSION

The order of merit of some ceramic bodies evaluated in thermal shock was experimentally established according to the method described in reference 5. A correlation of thermal-shock parameter with experimental results is presented in table I.

With the exception of the tensile strengths of the materials at 1800° F, which were determined at this laboratory according to the methods described in reference 5, the physical properties of these materials were obtained from literature. In some instances, where experimental data were unavailable for a particular body, approximate values were determined from data that were available on similar materials. These cases are noted in table I. For example, data on the stabilized zirconium dioxide ZrO_2 containing 6-percent calcium oxide CaO were unavailable; data for pure ZrO_2 were consequently used in evaluating the parameter for this material.

The evaluation of the parameter $\frac{ks}{\alpha E}$ for five materials indicates that increase in resistance to fracture by thermal shock accompanies increase in $\frac{ks}{\alpha E}$. In table I, the value for titanium carbide TiC is 12,560 and for stabilized zirconium dioxide ZrO_2 plus calcium oxide CaO, which is much poorer in thermal-shock resistance, the value is 562.

An examination of the type of fracture sustained during the thermal-shock evaluations shows that all the materials listed failed during the cooling part of the cycle with the exception of the stabilized zirconium dioxide, which failed in shear during the first cycle. The shear failure, as observed on stabilized zirconium dioxide, results from compression produced during the heating portion of the shock cycle. In general, if the stress for fracture in shear is less than one-half that necessary for failure in tension, a shear failure occurs before tensile failure. The tensile strength for stabilized zirconium dioxide at 1800° F is 5400 pounds per square inch (table I); this value is used in the determination of $\frac{ks}{\alpha E}$. Actually, because the stabilized zirconium dioxide disks failed in shear, the value of s should be an equivalent tensile strength (somewhat less than the value of 5400 lb/sq in. shown in table I) equal to twice the shear strength.

If a shape and size factor were included in the parameter, a dimensionless number might be obtained that would permit predicting the resistance of a ceramic material to fracture by thermal shock in any shape or size.

CONCLUSION

The resistance of a ceramic material to fracture by unsteady-state heat flow (thermal shock) can be correlated with a material parameter that is qualitatively determined from the product of thermal conductivity and tensile strength divided by the product of linear coefficient of thermal expansion and ductility modulus. Additional experimental data are required on convection heat-transfer coefficients before a quantitative prediction of the failure of ceramic materials by thermal shock can be made.

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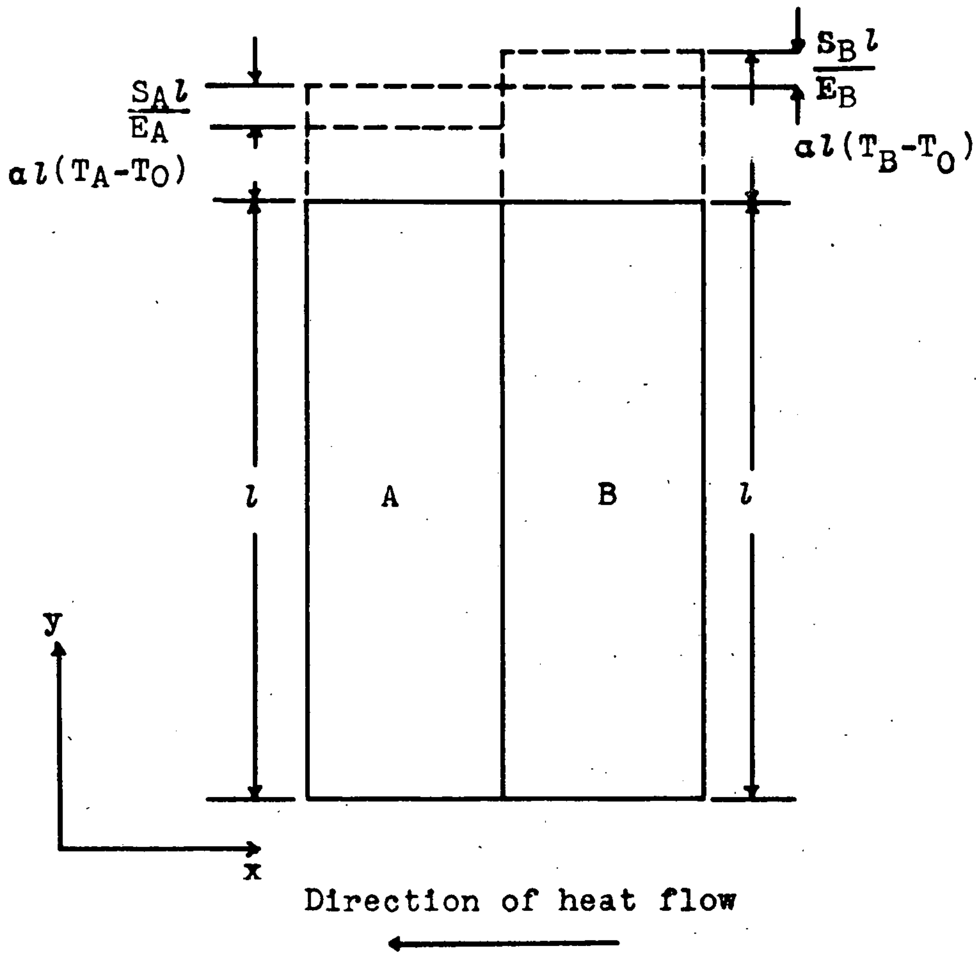
TABLE I - CORRELATION OF MATERIAL PROPERTIES WITH RESISTANCE TO FRACTURE BY THERMAL SHOCK

Order of merit of materials evaluated in thermal shock	Thermal shock (cycles before failure) Temperature, (°F)				Coefficient of thermal expansion, $\alpha \times 10^{-6}$ (in./in./°F)	Thermal conductivity, k (Btu/(hr)(sq ft)(°F/in.))	Ductility modulus at 1800° F, $E \times 10^7$ (lb/sq in.)	Tensile strength at 1800° F, s (lb/sq in.)	$\frac{ks}{\alpha E}$
	1800	2000	2200	2400					
Titanium carbide (TiC)	25	25	25	21	5.0	$\approx 240^a$	6.0^a	15,700	12,560
Beryllium oxide (BeO)	25	3	--	--	4.8	104	4.28	6200	3139
Zircon ($ZrSiO_4$)	1	--	--	--	2.5	11.6	2.4	8700	1682
Magnesium oxide (MgO)	$\frac{1}{2}$	--	--	--	7.7	16-40	1.24	3100	519-1299
94-percent zirconium dioxide (ZrO_2) + 6-percent calcium oxide (CaO)	0	--	--	--	5.5	14.3	2.5^b	5400	562

[a]Value for 80-percent titanium carbide and 20-percent cobalt.
[b]Value for pure zirconium dioxide.



E_A ductility modulus of element A, lb/sq in.

E_B ductility modulus of element B, lb/sq in.

l length, in.

S_A stress in element A, lb/sq in.

S_B stress in element B, lb/sq in.

T_A temperature of element A, $^{\circ}F$

T_B temperature of element B, $^{\circ}F$

T_O temperature of gas surrounding specimen, $^{\circ}F$

α linear coefficient of thermal expansion of specimen, in./in./$^{\circ}F$

Figure 1. - Elongation of two elements of body as result of temperature gradient $\frac{dT}{dx}$ in body.

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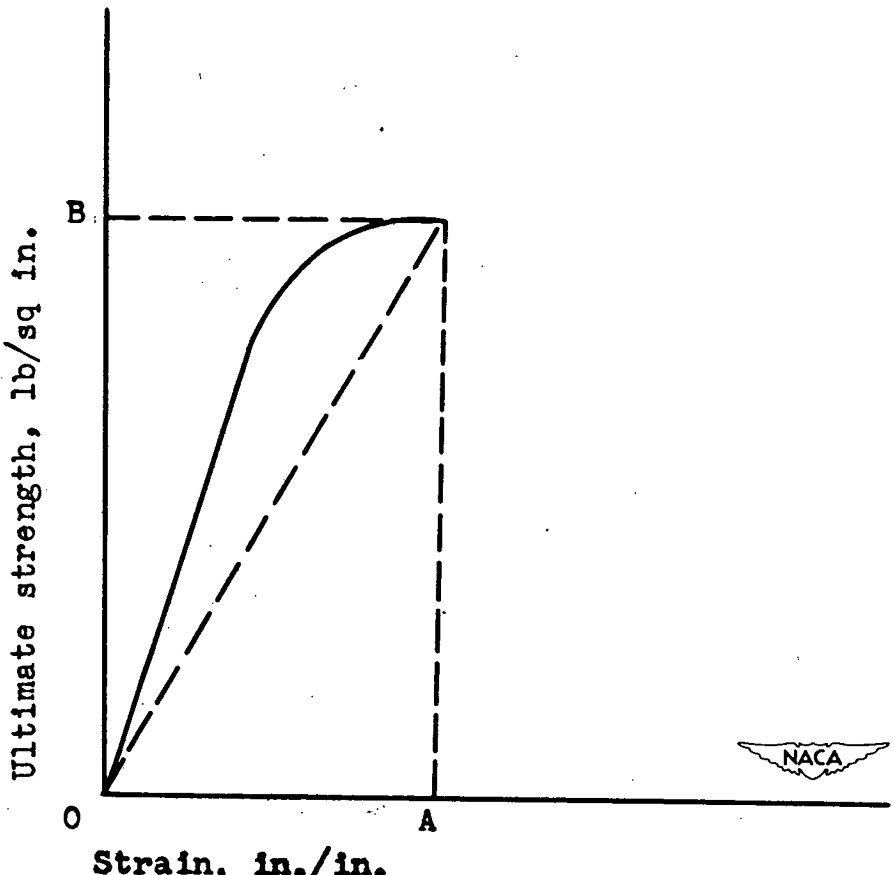

Ductility modulus $E = \frac{\text{Ultimate strength}}{\text{Strain}} = \frac{OB}{OA}$

Figure 2. - Relation between ductility modulus E and stress and strain at failure.

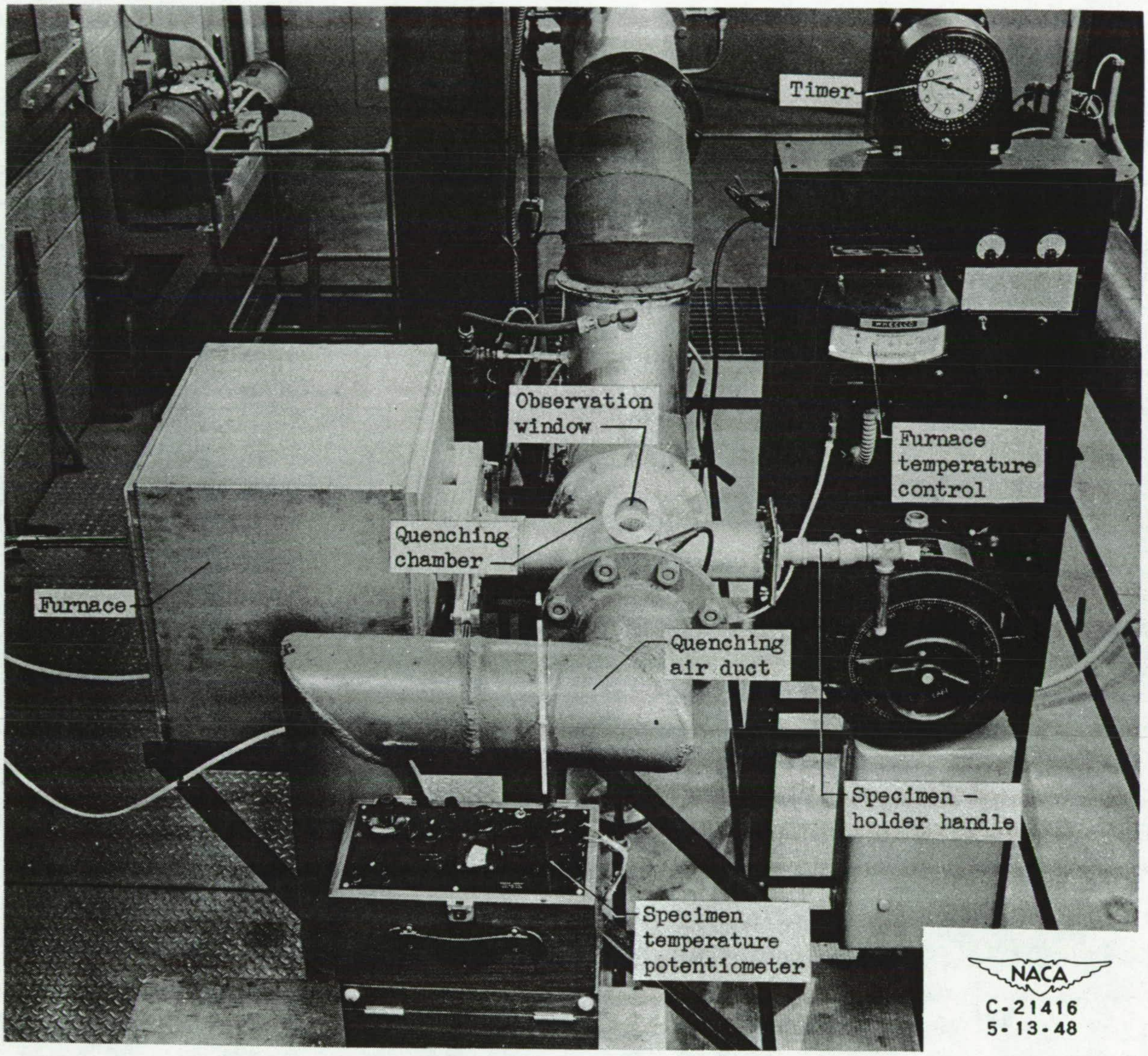

Figure 3. - Apparatus used for determinating resistance of materials to fracture by thermal shock.