CHARACTERISTICS OF THE HIGH TEMPERATURE MECHANISMS OF CREEP AND RECOVERY IN GRAPHITE

Contract No. DA-91-591-E.U.C.1759

F.T.R. No. 1

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Bletchley,
Buckinghamshire,
England.

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ABSTRACT

The report summarizes the first year's work carried out on an investigation into the high temperature mechanisms of creep and recovery in graphite. This work has been devoted to the building of an experimental apparatus capable of exerting creep loads in the temperature range 2000°C - 3000°C. A full description of the apparatus is given and its further potential discussed. Detailed figures are included on the labour expended together with a summary of property acquired for use on the contract.

A. Summary and Analysis of Work Performed

The programme, throughout the period covered by this report, has been devoted to the design and construction of a creep apparatus. This machine is designed to apply torsional creep loadings to 1" diameter tubes in a temperature range up to 3000°C. The equipment is now ready for use, and the results obtained with it will be contained in subsequent reports. A description of the apparatus is given below together with an assessment of its potentialities.

i) Type of test and specimen

A simple stress system in which the stress for a constant load remains constant throughout the life of the test is achieved by applying a constant torque to thin-walled tubes. The stress may also be sensibly regarded as constant across the thickness of the wall where this thickness is small in comparison with the tube diameter. The specimens have a tube diameter of 1.000" with a wall thickness of 0.060". Three factors prevent a thinner wall being used. Firstly, the wall thickness would, in some graphites, approach the grain diameter, thus leading to unrepresentative results.

Secondly, the load for a given stress is reduced, which means that the absolute variation in the load during a test must be reduced for a given percentage variation on a set stress. Thirdly, graphite is brittle, and the machining of long thin-walled specimens would be difficult, and the uniformity along the gauge length consequently doubtful.

The specimens are to be made from extruded rods to the dimensions shown in Fig. 1. The gauge length adopted is 1 ins. An increase in gauge length would yield increased sensitivity in strain measurement because of the larger strain produced. However, length has had to be limited to 1" as the power requirements to heat uniformly a longer length to very high temperatures become quite large. The same factor also restricts the diameter of the specimen. The density of off-cuts from each of the rods will be determined and they will also be examined micrographically. These results can then be compared with those from material taken from the gauge length after straining. This procedure will ensure a strict comparison without any assumptions regarding batch homogeneity.

ii) Heating Unit

A carbon resistance tube is placed concentrically around the specimen. Indirect heating is considered to be superior to direct heating of the specimen by passing a current through it. By this 'internal' method local 'hot-spots' are easily set up, giving erroneous load-elongation results for the average temperature of the specimen. The resistance tube, machined into a spiral element over the central 3" is heated by a low voltage, AC supply. The element is surrounded by three carbon and three molybdenum radiation shields. These fulfill the dual functions of conserving heat and protecting lower melting point components. The element and the shields are supported on water-cooled copper plates, two of these also acting as conductors. The whole unit is shown in Fig. 2. Power is obtained from a 240V single-phase AC supply. This is fed into a saturable reactor, the output voltage of which is controlled within the range 30 - 180V by the furnace temperature through a Bristol Dynamaster recorder/ controller. A secondary 10:1 step-down transformer reduces the power to the working voltage range of

3 - 18V. The maximum permissible continuous current is about 1,500A.

A 'Land' total radiation pyrometer having a minimum target diameter of 0.1" and specially designed for this apparatus, is focused on the specimen. Its output is measured and recorded by the Bristol Dynamaster instrument calibrated to work with the pyrometer. The Dynamaster then controls the output voltage of the saturable reactor to maintain constant temperature. The Dynamaster has four scale ranges covering the temperature range 1600°C - 3000°C. Thus a full scale deflection within any one range corresponds to a 400°C temperature change. Fine control should therefore be possible.

iii) Furnace Chamber and Vacuum Equipment

The chamber is constructed from $\frac{1}{2}$" thick steel plate with cooling coils soldered to the outside. Openings are provided for sighting two pyrometers and for the exhaustion of the chamber. Top and bottom plates are made from $\frac{3}{4}$" thick mild steel plate. The lower end of the specimen passes through the bottom plate (see Fig. 3) into the lower chamber. This contains the load and strain measuring units. The driving shaft providing the twisting couple passes through a Wilson seal on the base of this chamber.

D.J. Diefendorf (4th Carbon Conference) has shown an increase in the strength of graphite resulting from a degassing treatment of 24 hours at 1000°C under a pressure of 10^{-4} mm Hg. A pressure higher than 10^{-3} mm failed to give any increase in strength. In view of these results all specimens will be given a degassing treatment before testing. An oil-filled two-stage N.G.N. rotary pump backing an oil diffusion pump has been installed for this purpose.

Above 2000°C some volatilisation of the graphite occurs under vacuum. This can be suppressed by the admission of a low pressure of argon. Provision has been made for removing residual oxygen and water vapour from the argon. The argon supply will be directed on to the inside faces of the silica pyrometer windows to assist cooling and to provide a gas flow away from them to eliminate 'sooting' of the window. The vacuum pump will continuously evacuate the argon and the pressure will be maintained by adjustment of the input and exhaust gas flows.

iv) Water Cooling

It is essential in an apparatus of this nature to provide a 'fail-safe' water cooling system.

The copper plates and tubes carrying the heating assembly require a fast-flowing current of water to dissipate both the radiant heat falling upon them and the heat generated in them by the passage of the current.

A block diagram of the water system is given in Fig. 4. Water is pumped from an underground tank up through the rig and into an elevated storage tank. From thence it overflows back to the underground tank. A large fraction of the water pumped passes directly back to the underground tank via the by-pass line. Should the pump fail, the water would flow back from the upper tank through the rig and via the by-pass line to the underground tank. With the mains make-up water flowing into the upper tank, the flow from this tank would protect the rig in the event of pump failure. The initial loss of pressure automatically switches out the heating current.

The temperature of the water in all the pipes leaving the heating unit is continuously indicated on gauges.

v) Torsion Drive

The drive to the specimen is derived from a hydraulic rotary actuator situated immediately below the lower chamber. A shaft passing through a Wilson seal connects the actuator to the torque-load measuring unit.

The actuator is a single-vane reversing type and is supplied from a Keelavite 'Midget' power pack via a two-way electromagnetic reversing valve. This enables the load to be reversed from full on, through zero to full on in 15 milliseconds. Replacement of the manual reversing switch by an automatic unit would enable fatigue experiments to be carried out on the rig.

The power pack incorporates a micro-filter, a pressure gauge and a by-pass type valve for pressure regulation. This will control pressure to within ± 3 p.s.i. As a stress on the specimen of 1000 p.s.i. will require a hydraulic pressure of only 150 p.s.i., this pressure variation is felt to be unacceptable. It is proposed, therefore, to instal a second reducing valve coupled to a servo-motor operated by an error signal from the load measuring unit.

vi) Load Measuring Unit

A photograph is shown in Fig. 5. The driving shaft drives three identical steel arms placed at 120°C to each other. Near their outside ends these arms bear on knife edges. These knife edges are rigidly suspended from a collar fixed to the specimen. The weight of the collar is supported by a bearing. A rotary transducer measures the relative movement between the driving shaft and the collar. This deflection is directly proportional to the applied load.

The rotary transducer is supplied by an oscillator-demodulator unit manufactured by the Instrumentation Section of the College. The demodulated D.C. output is fed into a Blackburn Data Amplifier (maximum gain 1000) and then measured on a Blackburn Digital Voltmeter. As discussed above, it is proposed to use this signal to control the hydraulic load. As there is no rigid connection between the driving shaft and the collar, the specimen, on recovering, has only the low inertia of the collar to overcome.

vii) Strain Measuring Unit

The principle of this unit is shown in Fig. 6. The rotary transducer is identical to that used for load measurement and is served by the same oscillator-demodulator unit. By using the solid lower end of the specimen to transmit the load and by measuring its rotation directly there are no errors arising from movements between any coupled parts.

The maximum theoretical resolution of this system is high. A 1° movement of the specimen will produce a 1.75V signal from the transducer. Using the data amplifier and the digital voltmeter there is a maximum resolution of $5 \mu V$, corresponding to a specimen rotation of 3×10^{-6} degrees. It is unlikely that this order of resolution will normally be achieved owing to instabilities in the oscillator unit and other general mechanical considerations of the rig. Efforts will be directed to a maximum use of the high resolution offered by the equipment.

viii) Assessment of other potentialities

Although primarily designed for high temperature creep work, the apparatus can fulfill several other functions. The heating unit can in itself be used as a convenient heat treatment furnace, with a vacuum or a controlled atmosphere, for refractory metals. The introduction of an automatic reversing switch would enable torsional fatigue tests to be carried out at a maximum frequency of 33 c.p.s. over the whole range of temperatures and stresses intended for the creep work.

The apparatus should be capable of carrying out creep experiments of a high degree of accuracy. The maximum resolution of the strain measurements may be degraded by mechanical vibrations in the rig and by thermal fluctuations in the oscillator-demodulator unit. The former will be minimised as far as is practical. Changes in output of the oscillator unit are reflected in changes in a reference signal given by it which is to be monitored by the voltmeter. Therefore changes in the output of this unit will be known and due allowances made in the measurements.

B. Implications of results

The implications of this work remain as described in the original proposals for the research. Recent work at Cambridge University has led to the conclusion that dislocation climb cannot be the controlling mechanism for the creep of graphite at high temperatures. This would imply that a different mechanism to that in operation in metals is involved, and gives an added incentive for the current investigation.

C. Personnel

The programme has been carried out throughout the whole year under the general direction of Professor A.J. Kennedy. The work has been led by a graduate scientist (Dr. A. Younger) assisted by a research technician (Mr. R.C. Walding). Dr. Younger has worked on the project for the complete year and Mr. Walding for the last ten months.

D. Man-Hours, Property acquired, etc.

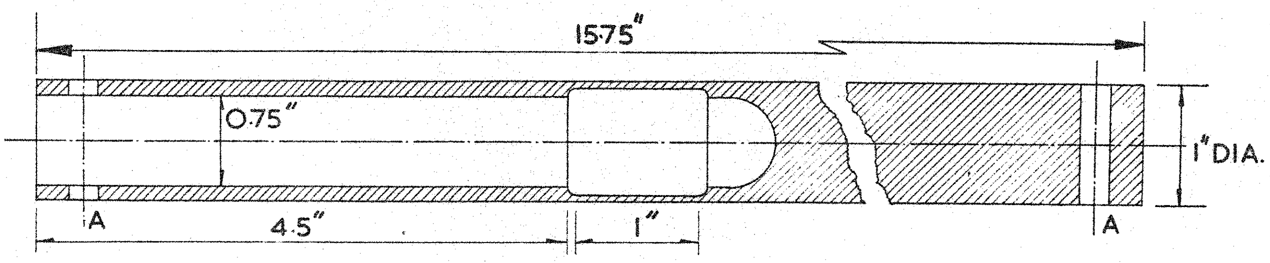
(i) Hours

Dr. A. Younger	1,800	hours	approx.
Mr. R.C. Walding	1,700	"	"
Main Workshops personnel	3,700	"	"

(ii) Property acquired for use on contract work.

	£
Saturable Reactor	315
Temperature Controller	325
Pyrometer	93
Step-down transformer	132
Water Pump	60
Vacuum Pump, gauges, valves, 'O' rings, etc.	139
Molybdenum Shields	37
Hydraulic power pack, actuator valves and pipelines	219
O.F.H.C. copper for internal conductors	52
Digital Voltmeter with Data Amplifier	1,045
Miscellaneous electrical and electronic items	121
Miscellaneous small mechanical parts and tools	19
	£2,557

plus a large quantity of electrical and general engineering fittings, tools and metal stock drawn from College Stores.



'A'. REAMED HOLE 0·250" DIA. 0·406" FROM END OF SPECIMEN

FIG. I. TORSION SPECIMEN

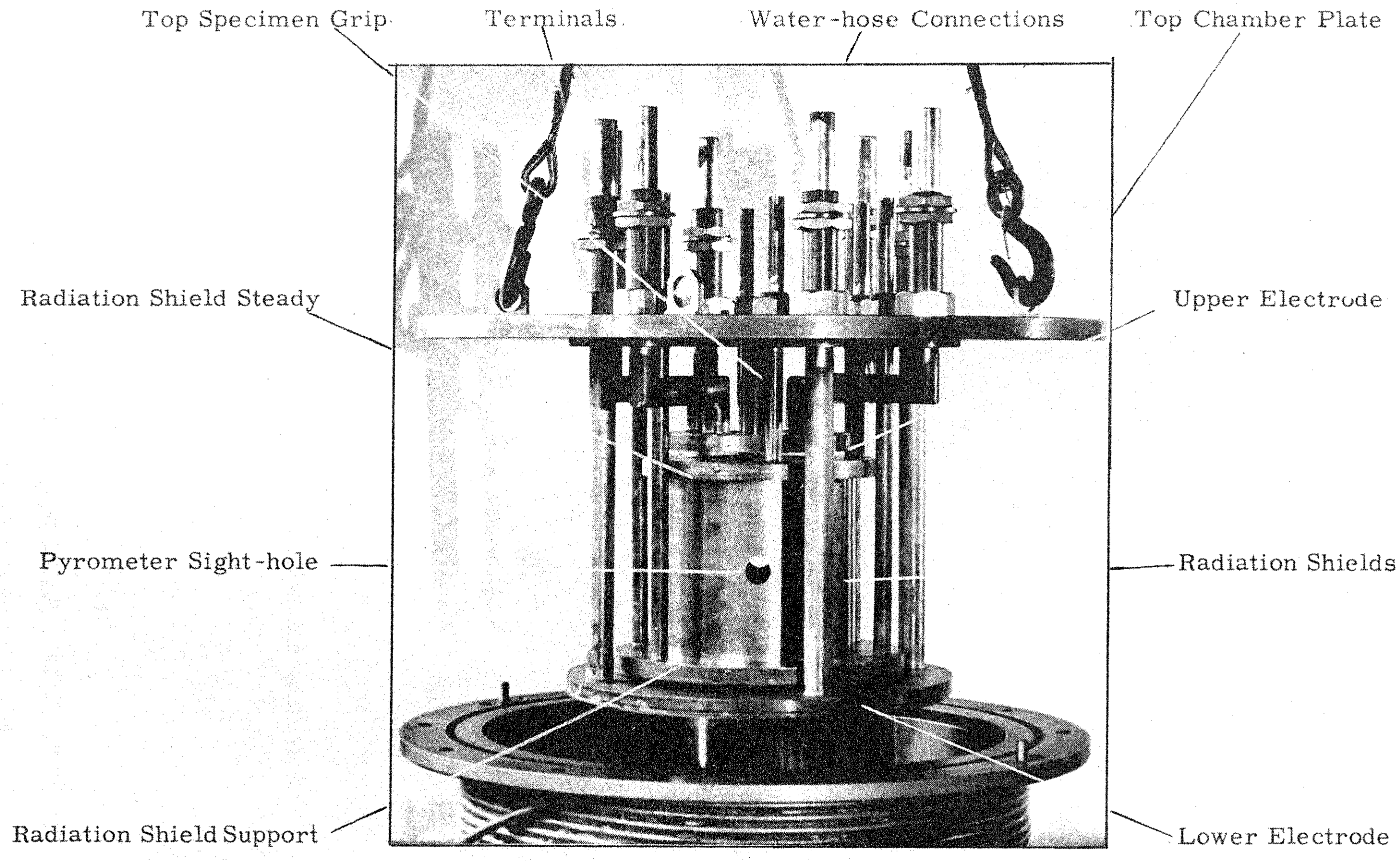


FIGURE 2. HEATING UNIT

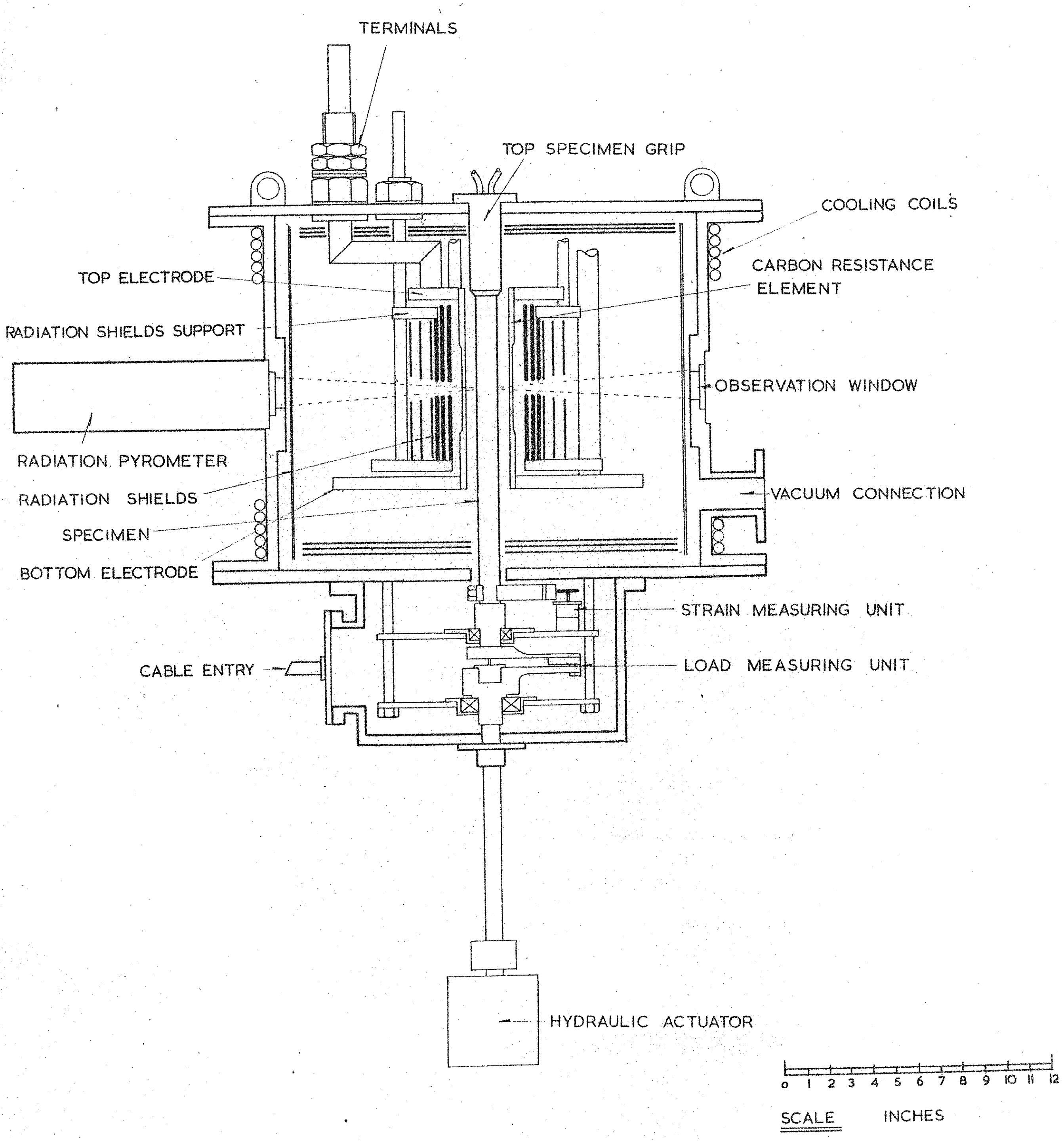


FIG.3. DIAGRAM SHOWING GENERAL ARRANGEMENT OF UPPER AND LOWER CHAMBERS OF TORSION RIG

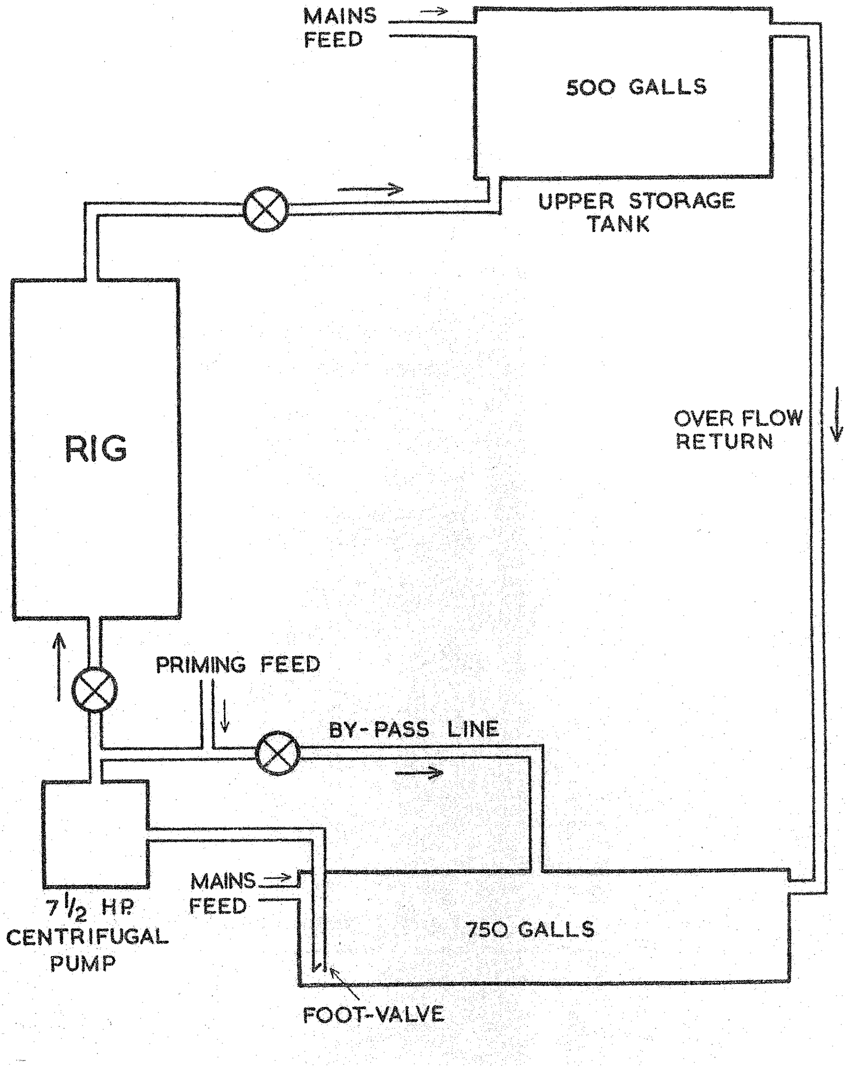


FIG.4. FLOW DIAGRAM OF WATER COOLING SUPPLY

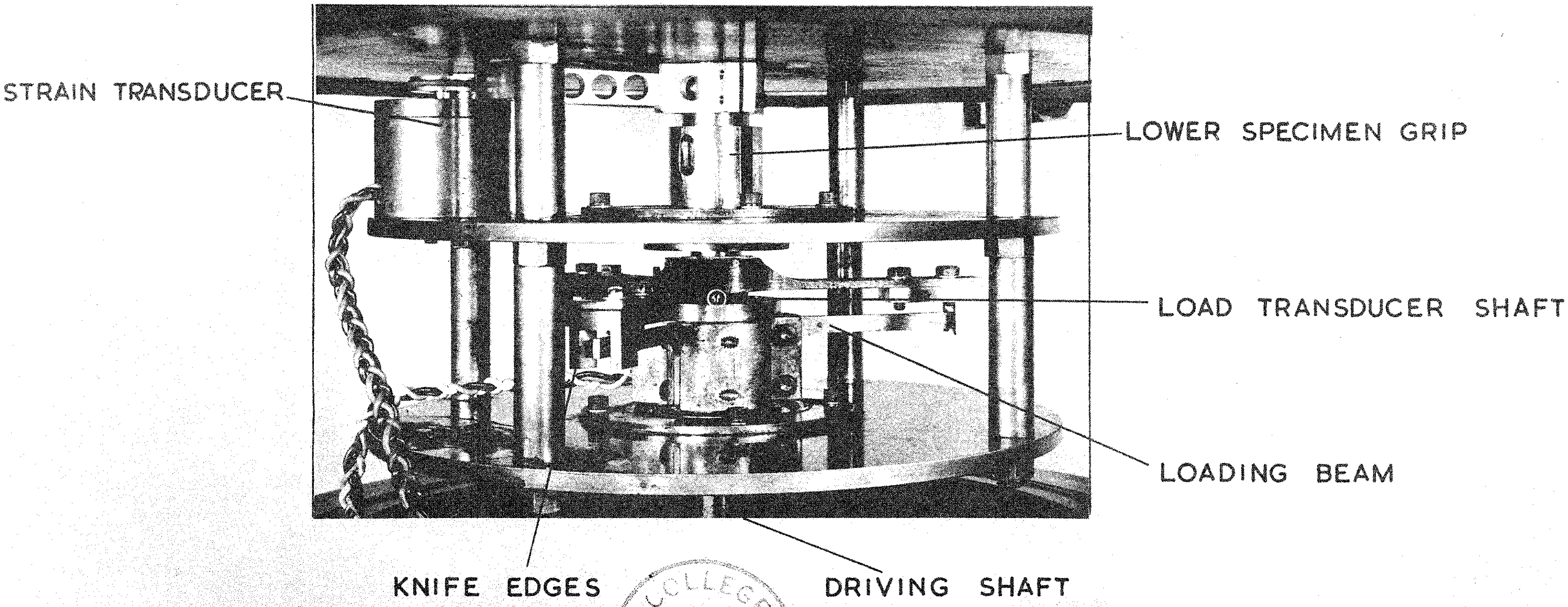


FIG. 5 LOAD AND STRAIN MEASURING UNIT

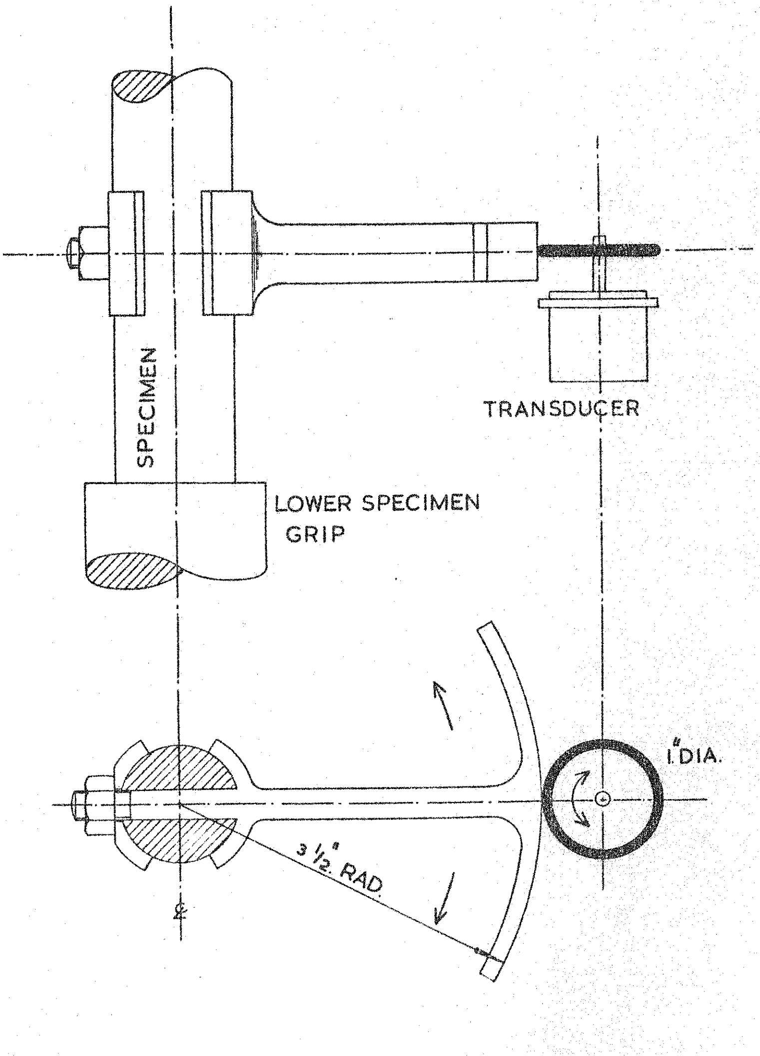


FIG.6. STRAIN–MEASURING MECHANISM