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A METHOD OF SPECIMEN CORROSION PROTECTION FOR HIGH TEMPERATURE CREEP TESTING

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Abstract--The determination of mechanical properties of materials at elevated temperatures presents difficulties, particularly when the material to be tested is subject to oxidation. Various methods have been employed to permit the evaluation of high temperature creep properties. The method described in this paper was developed on the basis of modifications of a technique developed for the protection of high temperature fatigue specimens. The method involves encasing the creep specimen in a flexible capsule which is capable of withstanding exposure to the atmosphere for extended periods at temperatures up to 1000° C.

Extensive testing of materials such as uranium and tantalum has provided the basis for claims relative to the effectiveness of this technique.

INTRODUCTION

Interest in high temperature properties of materials which in themselves do not possess the ability to withstand corrosion of the atmosphere has been stimulated by current nuclear applications. The inadequate means of conventional testing equipment to provide the required oxidation protection for these materials has without doubt retarded advancements in this field. In creep and fatigue tests, where the periods of testing are usually long, the problem of oxidation is most severe. A highly satisfactory means of oxidation protection for fatigue specimens was developed by Bohn and Murphy,[1] and it is from this work that this method of protection for creep specimens has evolved.

Oxidation is obviously prevented if oxygen is not allowed to come in contact with material under test. This implies that an oxygen tight barrier be placed between the specimen and the oxygen laden atmosphere. The barrier may be placed (a) surrounding the appropriate components of the testing machine; (b) on the surface of the specimen; or (c) surrounding, but not in intimate contact with the specimen.

The most common approach taken in the design of oxidation protection equipment for high temperature creep studies has been based on (a). The unusual cost and complexity of operation and maintenance of this type of equipment poses obvious disadvantages.

Limited success was obtained using method (b) for creep applications prior to this investigation. The method involved electroplating copper, nickel and silver, alone and in multiple layer combinations, onto the specimen. Diffusion of oxygen through the plating at higher temperatures caused scaling and reduced the effectiveness of the technique.

A technique for oxidation protection based on (c) was developed and proven satisfactory for temperatures up to 1000°C. The technique

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(1) Bohn, J. R. and Glenn Murphy, High-temperature fatigue testing with application to uranium. American Society for Testing Materials Bulletin, 234:57 (1958).

provided a dependable low maintenance system which utilized commercially available basic high temperature creep testing equipment. The method of protection involved encasing the creep specimen in a flexible capsule capable of withstanding prolonged exposure to the atmosphere at high temperatures. Provisions were made to attach extensometer linkages through the capsule walls to the specimen gage points. A compound system of gage points was used to render the desired coupling.

ENCAPSULATION

The capsule consisted of several sections of bellows welded to machined parts which comprised the gage point mounting rings and specimen end-adaptors. The build-up of the various components is shown in Fig. 1. An assembly drawing of the capsule is given in Fig. 2, which illustrates in more detail the functions of the various components and provides also an index to their description. The details of the components are given in Figs. 3, 4 and 5. Single-ply seamless stainless steel bellows were used. In several minor forming operations the end convolutions on each bellows were formed to match the corresponding weld flanges on the appropriate machined parts. The creep specimen used in this investigation is shown in Fig. 6. Overall capsule dimensions were based on this specimen.

Fabrication of the capsule was accomplished readily using shielded-arc welding techniques with the aid of specially designed jigs and fixtures. The procedures and apparatus used in fabrication

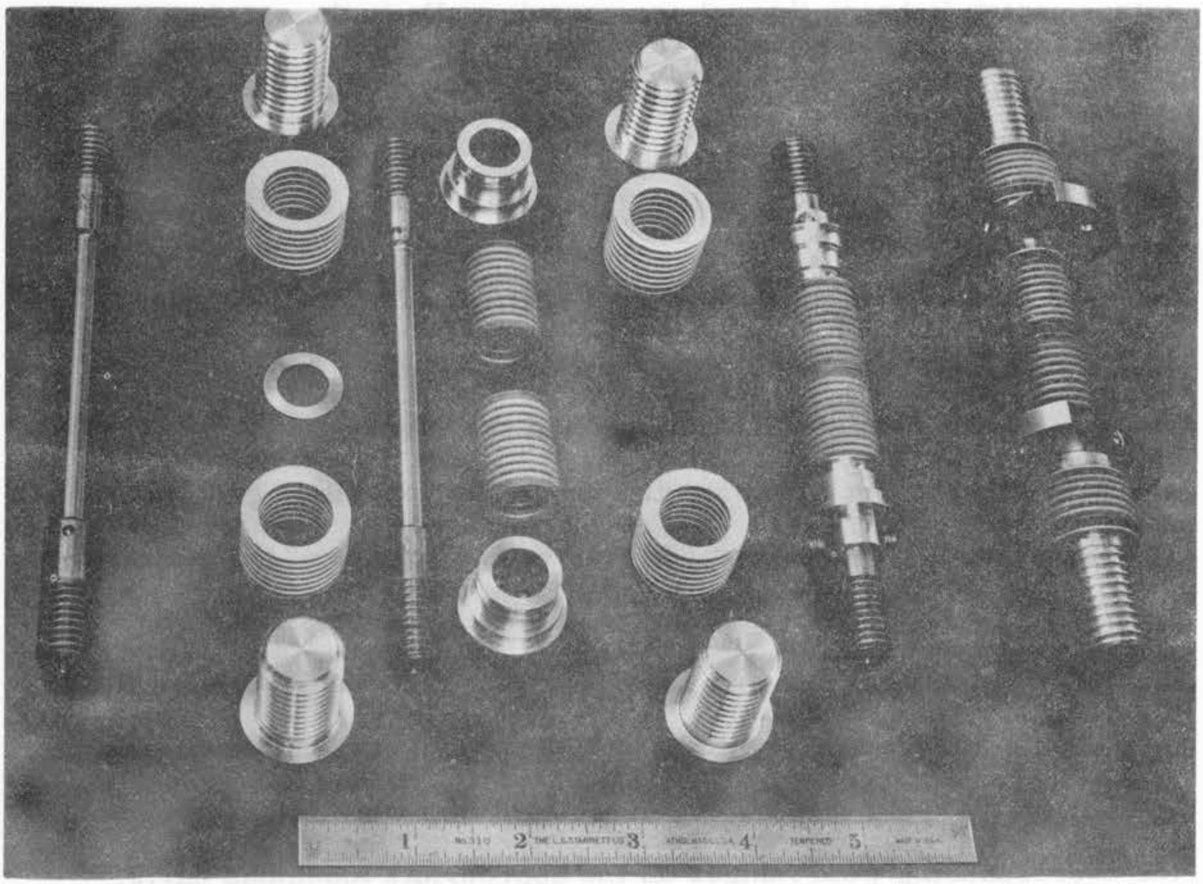


Fig. 1 - Build-up of components.

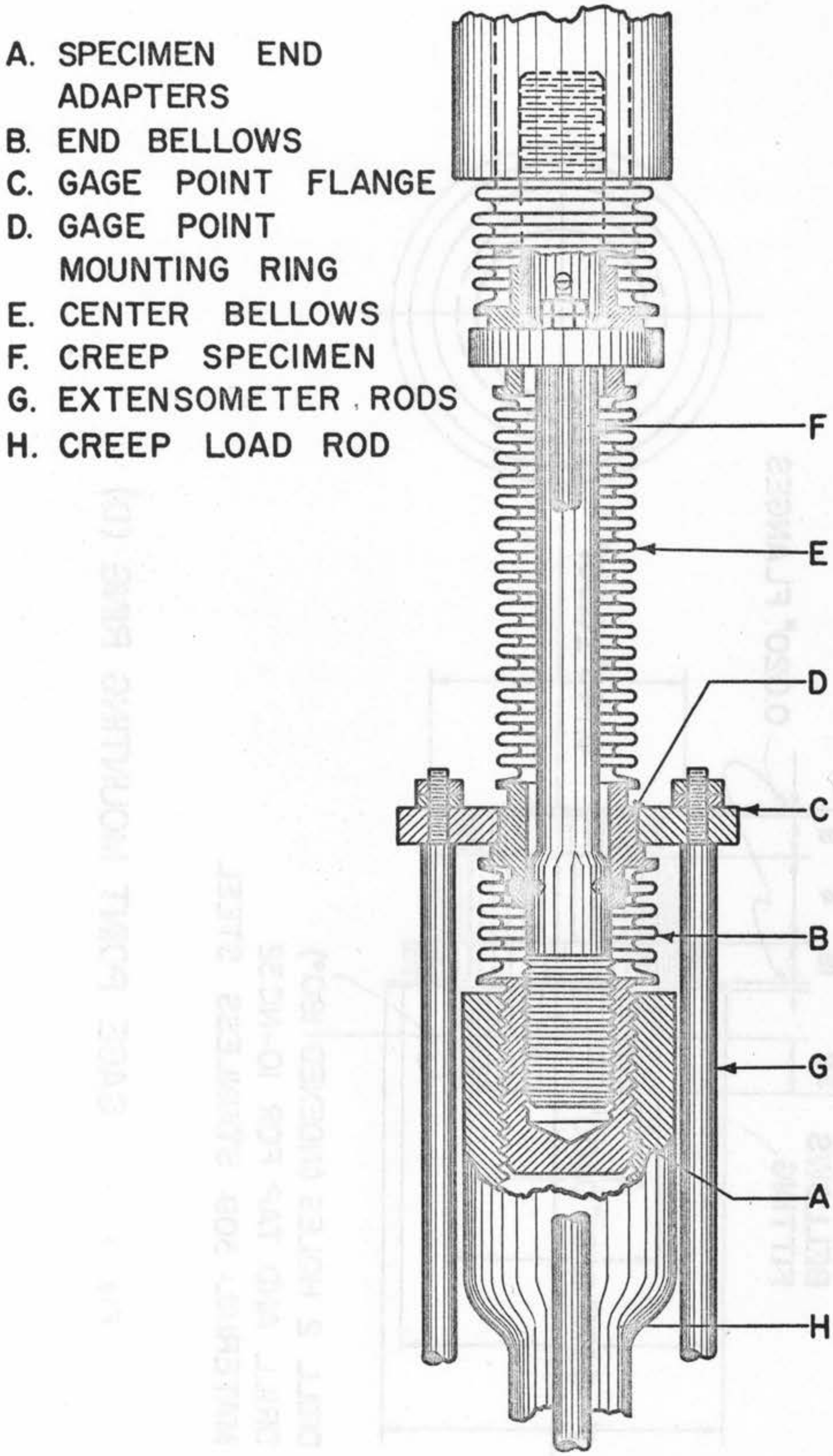
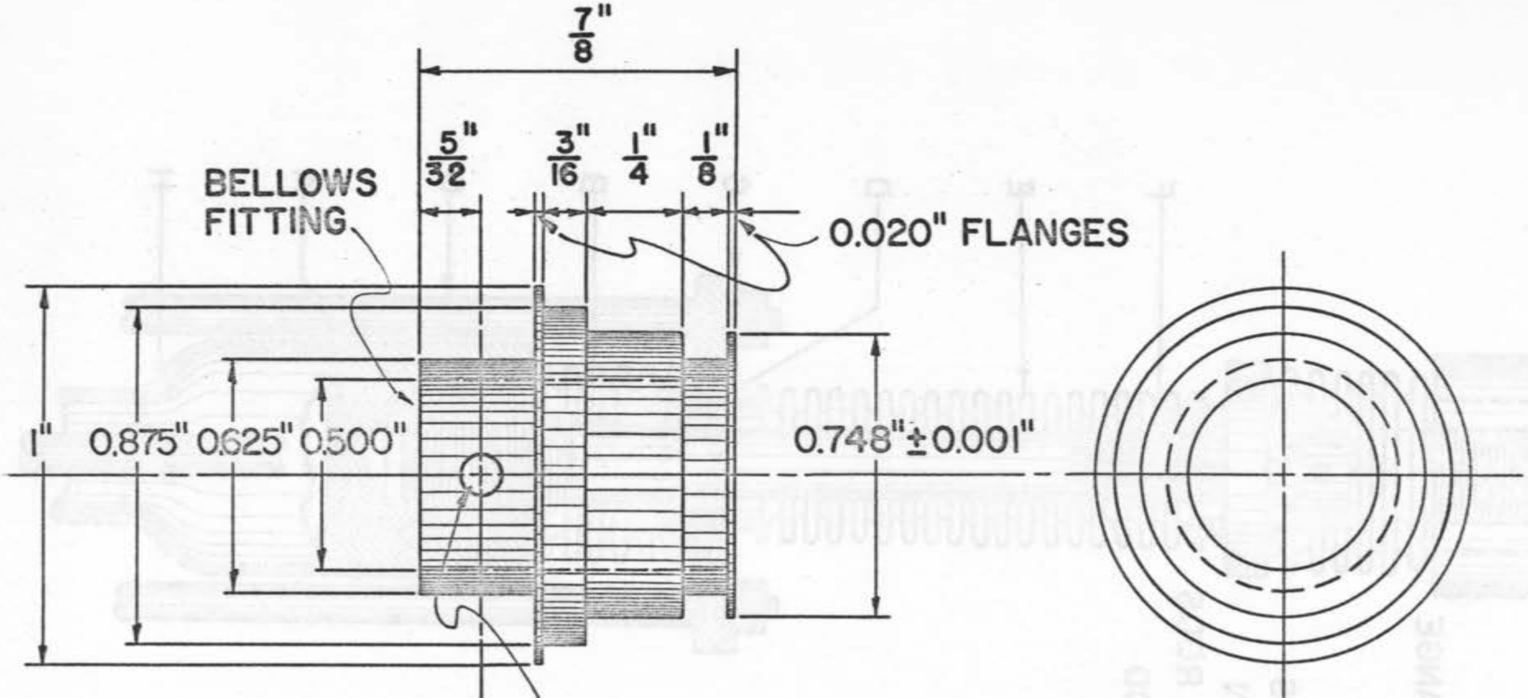


Fig. 2 - Assembly drawing of the flexible capsule.



DRILL 2 HOLES (INDEXED 180°)
DRILL AND TAP FOR 10-NC32
MATERIAL: 309 STAINLESS STEEL

Fig. 3 - **GAGE POINT MOUNTING RING (D)**

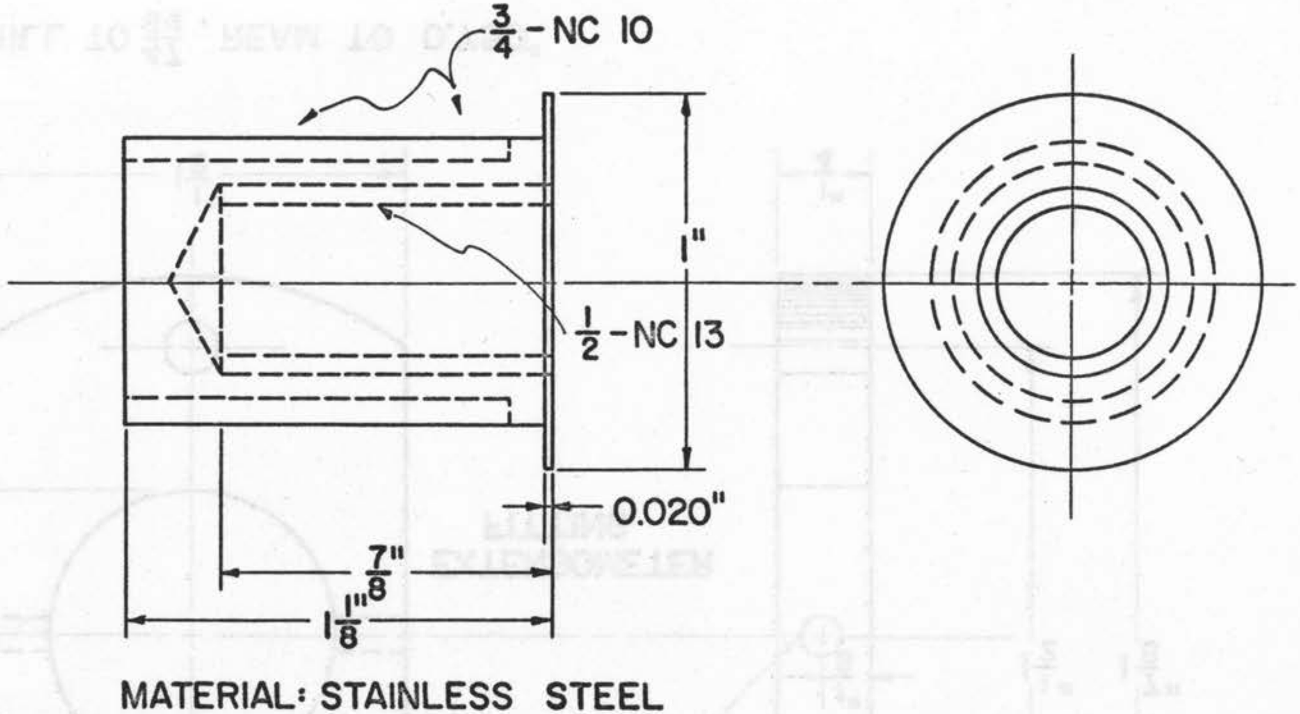
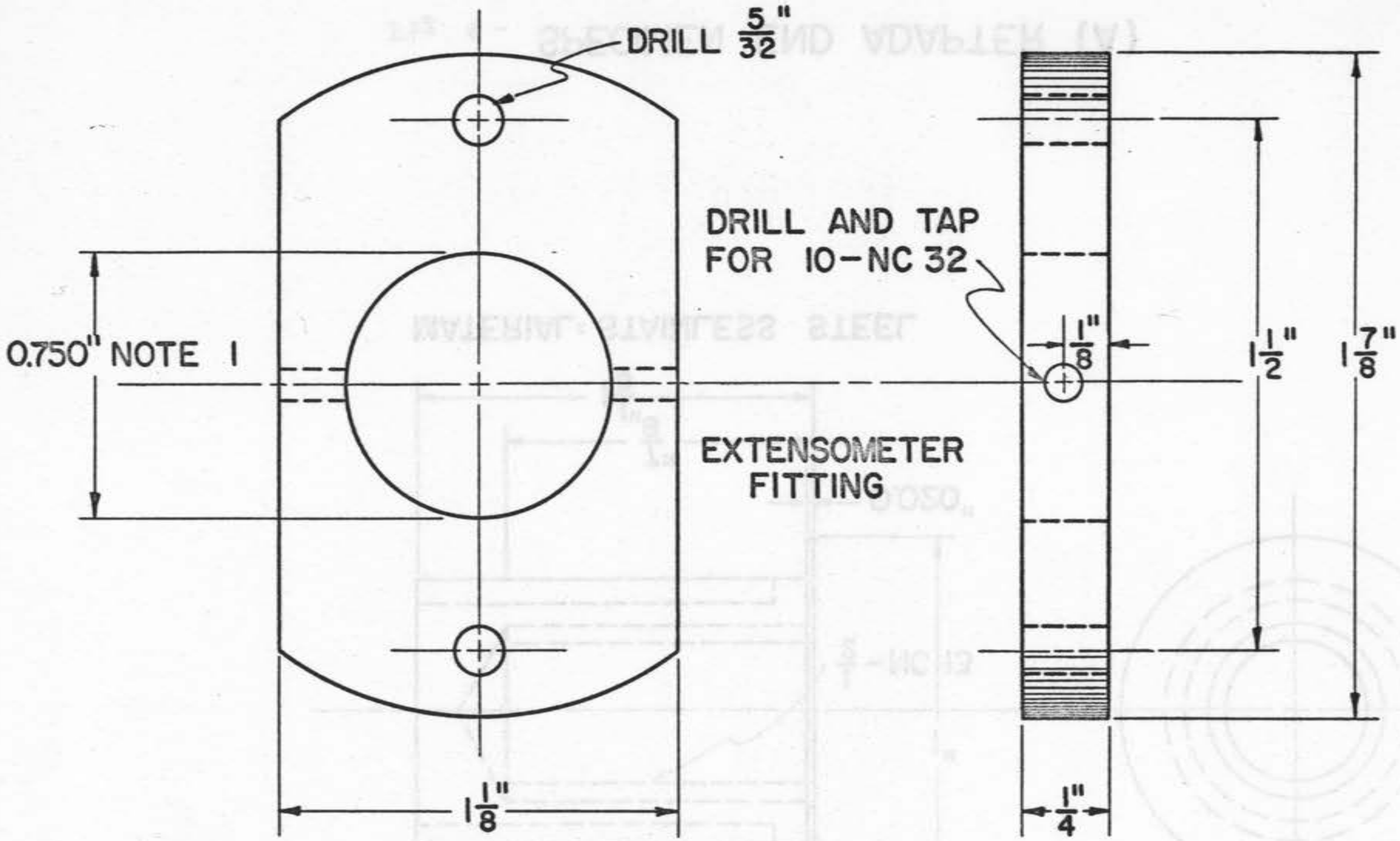
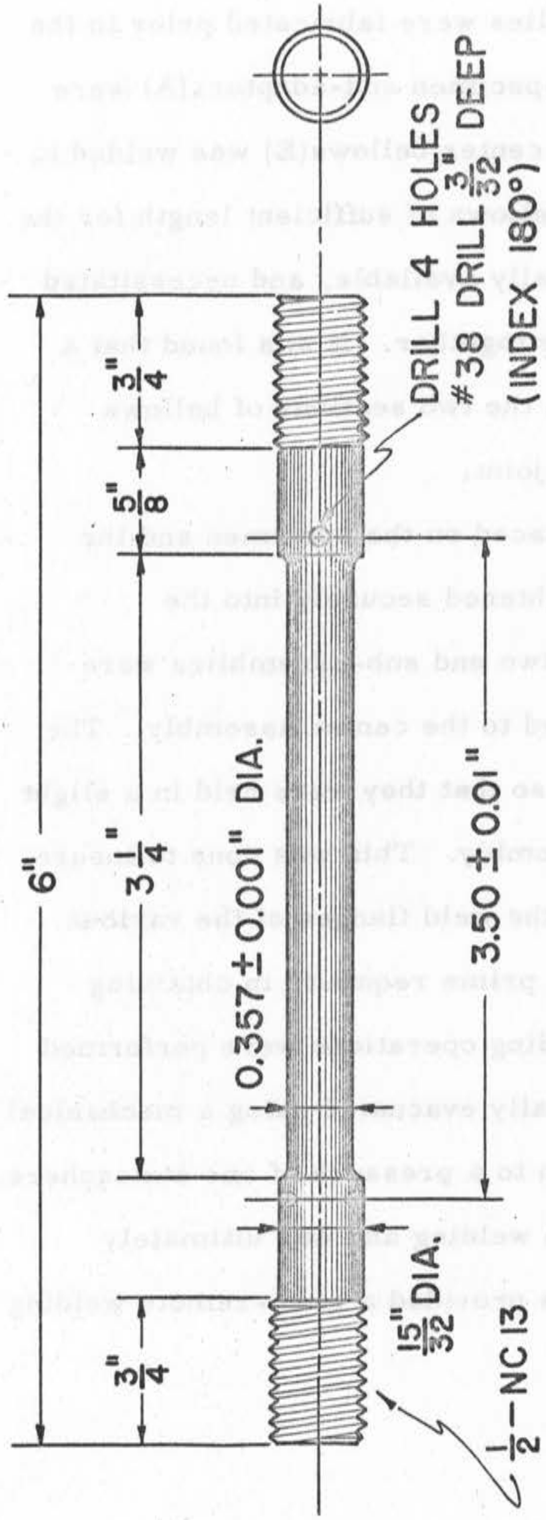


Fig. 4 - SPECIMEN END ADAPTER (A)



NOTE 1: DRILL TO $\frac{47}{64}$, REAM TO 0.750".

Fig. 5 - GAGE POINT FLANGE (C)



MACHINED FROM 1/2" DIAMETER RODS 6" LONG

Fig. 6 CREEP SPECIMEN (F)

were discussed in detail by Bohn and Murphy[2] thus the only concern here will be the necessary description.

Several pre-welded sub-assemblies were fabricated prior to the final encapsulating operation. The specimen end-adaptors(A) were welded to the end bellows(B) and the center bellows(E) was welded to the gage point mounting rings(D). Bellows of sufficient length for the center assembly were not commercially available, and necessitated welding two short sections of bellows together. It was found that a thin washer(0.020 in) placed between the two sections of bellows aided in producing a uniform welded joint.

The center sub-assembly was placed on the specimen and the inner gage point set-screws were tightened securely into the specimen gage point holes, then the two end sub-assemblies were screwed onto the specimen and welded to the center assembly. The lengths of the bellows were adjusted so that they were held in a slight state of compression in the final assembly. This was done to insure a good mechanical coupling between the weld flanges of the various components, which was found to be a prime requisite in obtaining secure welded joints. All of the welding operations were performed in a welding chamber which was initially evacuated using a mechanical fore pump and then purged with argon to a pressure of one atmosphere. The argon acted to support the arc in welding and was ultimately sealed in the capsule. The apparatus provided a semi-remote welding

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(2) Ibid.

operation, in that the work was positioned and rotated by a constant speed drive mechanism, whereas, the electrode was held in a fixed position. Positioning the electrode was accomplished from the exterior of the chamber. Fig. 7 is a view of the welding apparatus.

PROTECTION

Several disadvantages are inherent in this method of oxidation protection. They include: (1) the uncertainty of leak-tightness of capsule welds, and (2) the exact determination of the specimen temperature.

The first has not presented any real difficulty in this investigation, since visual inspection of the welds usually indicated whether or not a tight seal had been achieved. Additional leak checks were occasionally made when prompted by doubt in visual inspection. One method involved replacing the welded assembly in the weld chamber and over-pressuring the chamber with argon to about 10 psig. After approximately one hour the assembly was removed and immersed as rapidly as possible in alcohol. Pin-hole leaks, if present, were detected by the appearance of bubbles. Another method employed was to evacuate the chamber, with the capsule included, immediately after welding. If the pumping time to obtain the minimum fore pump pressure was abnormally long, it was assumed that a capsule leak was present.

Experiments were performed using a modified capsule to determine the variation of specimen temperature as a function of

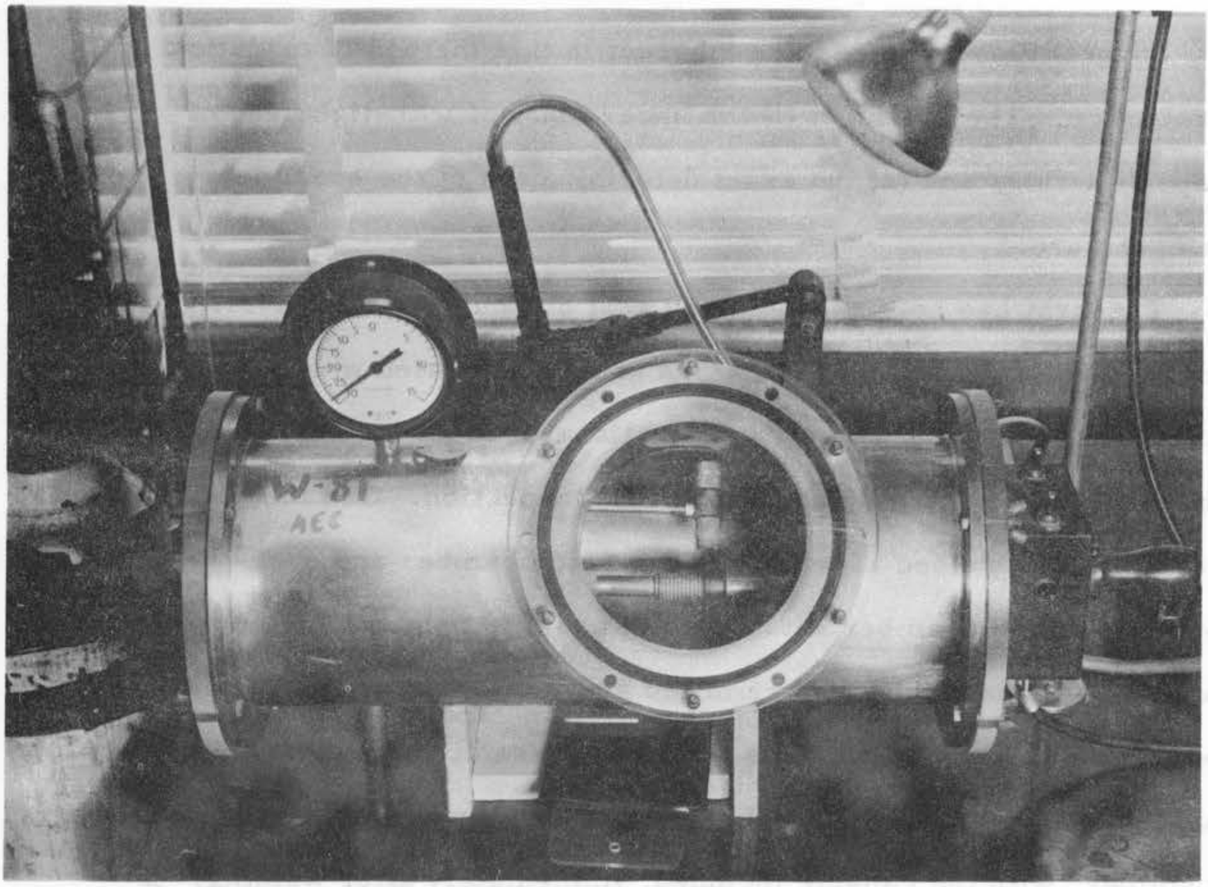

Fig. 7 - Welding chamber.

external capsule temperature. Provisions for thermocouple temperature measurements were made through seals in the capsule walls. As might be expected, the magnitude of the temperature difference varied as a function of the test temperature.[3] The observed difference between the specimen and external capsule temperatures at 800°C did not exceed $\pm$ 5°C; however, it is suggested that the appropriate temperature calibrations be made for the particular conditions of testing desired.

CONCLUSIONS

From the investigation reported it is concluded that encapsulation is a practical and effective means of protecting creep specimens to be tested at high temperatures. The method of protection is limited temperature-wise only by the temperature restrictions of the material used in construction of the capsule and the testing machines.

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(3) Hammel, R. L. Ames, Iowa. Private communication. 1959.