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LUCAS HEIGHTS

THE EFFECT OF NEUTRON IRRADIATION ON BERYLLIUM METAL

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ABSTRACT

This report summarises all the results obtained to date from a programme on the effects of neutron irradiation on the properties of beryllium metal. Results are presented on changes in density and mechanical properties in material fabricated by various routes and irradiated to fast neutron doses from 10^{19} nvt to 6×10^{20} nvt and at temperatures in the range 75°C – 700°C. Summaries of electron microscopy observations and electrical resistivity measurements, which are reported in more detail elsewhere, are also given.

It is concluded that all the observed property changes can be interpreted in terms of the distribution of helium which is produced by fast neutron transmutation reactions in beryllium and that damage due to defect production is negligible for irradiation temperatures of 75°C and above. Density changes due to helium bubble formation are shown to be very small but serious deterioration of mechanical properties can occur. The mechanical property changes and the distribution of helium are shown to be very dependent on material history and on the irradiation temperature. The standard Lucas Heights hot extruded material is shown to retain good mechanical properties for irradiation temperatures above 550°C but serious loss of low temperature ductility is found to occur for irradiation temperatures below 500°C, particularly in the range 300 – 500°C. It is concluded that nucleation of gas bubbles at precipitate particles is the only satisfactory explanation of the wide variations in behaviour of beryllium metal fabricated by various methods.

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1. INTRODUCTION

1.1 Radiation Effects in Beryllium

Beryllium metal is of interest as a moderator, canning material or fuel dispersant in various nuclear reactor systems. A research programme on radiation effects in beryllium metal has been pursued at Lucas Heights and this report summarises the work carried out to date. Because beryllium is no longer under consideration for the Australian Atomic Energy Commission's H.T.G.C. reactor study, this programme has been severely curtailed; however some further work on a limited scale is still in progress and will be reported later.

There are two mechanisms by which neutron irradiation can affect the properties of beryllium metal. Firstly fast neutrons can cause displacements of atoms from their normal lattice positions resulting in the formation of interstitial atoms and vacancies. These defects will disturb the regularity of the metal structure and hence affect any properties which depend on this regularity. At elevated temperatures the defects will anneal by diffusion and recombination; in the case of beryllium, as is shown later, they appear to be almost fully annealed at temperatures of 50 – 100 °C and above, so from a reactor technology point of view, this mechanism is of little interest.

The second mechanism arises from the fast-neutron transmutation reactions in beryllium which result in the formation of helium and tritium. These reactions are:

- $Be^9 + n \rightarrow 2\,He^4 + 2n$, which has a neutron energy threshold of 1.85 MeV.
- $Be^9(n, \alpha)\,He^6 \xrightarrow{\beta^-} Li^6$ which has a threshold of 0.71 MeV.

The Li-6 formed in the second reaction is almost entirely transformed to helium and tritium by thermal neutron capture. The net effect of the reactions is to produce an atomic dispersion of helium and tritium in the metal. At low temperatures these atoms will remain in enforced solid solution and produce property changes normally associated with solid solutions.

At elevated temperatures, diffusion may occur leading to the nucleation and growth of gas bubbles in the metal; these bubbles can cause volume changes and affect the mechanical properties of the metal. The work done so far both at Lucas Heights and in overseas laboratories indicates that the build-up of helium and tritium can result in significant property changes and may be the limiting factor in determining the performance of beryllium in a reactor core.

1.2 The Lucas Heights Programme

The research programme at Lucas Heights on irradiation effects in beryllium metal has had two main aims:

- To determine the effect of neutron irradiation on the properties of beryllium significant in reactor design and the effect of fabrication and structural variables on these property changes. The ultimate aim of this work would be to specify limits of temperature, neutron dose, stress, etc., within which beryllium could be used satisfactorily.
- To gain an understanding of the mechanisms which produce the observed property changes.

The second aim is essential to the success of the first and both are of necessity very closely integrated in the experimental programme. An understanding of the mechanisms involved is required for proper assessment of the results on property changes, for extrapolation of these results to different conditions, and to open up the possibility of developing material with greater resistance to detrimental property changes.

The programme involved the investigation of the effect of the following variables on the property changes:

- Fabrication method.
- Impurities, particularly oxide content.
- Irradiation temperature.
- Irradiation dose.

The following measurements and examinations were made to determine the effects of irradiation

- Density and dimensions.
- Mechanical properties including hardness and 0.1 per cent. proof stress, U.T.S., elongation, and reduction of area in tensile tests.
- Microstructure using both optical and electron microscopy.
- Electrical resistivity.
- Stored energy.
- Long wavelength neutron scattering.

Specimens of various sizes required for the above measurements were irradiated at either pile temperatures (75 – 100 °C) or at elevated temperatures (400 – 800 °C). Although the pile temperature irradiation (75 – 100 °C) is not directly relevant to reactor technology, annealing studies of the behaviour of material irradiated at these temperatures can provide much useful information; as these irradiations are much simpler to carry out than the high-temperature irradiations, they have been used extensively in this programme.

Some preliminary results from this programme were published previously (Hickman 1961) and detailed reports on some of the individual property measurements have been issued or prepared (Chute 1963; Svenson and Hickman 1963). Some irradiation experiments on beryllium metal have been carried out at Lucas Heights under contract to the U.K.A.E.A. and have been reported separately (Hickman et al. 1962; Hickman et al. 1963 unpublished).

This report describes the experimental methods used in the programme and summarises and discusses all the results obtained to date, with particular emphasis on the mechanical property studies.

2. EXPERIMENTAL METHODS

2.1 Materials

The material used for most of the work was fabricated from leached Pechiney electrolytic powder by direct extrusion at 1050 °C as described by Wright and Silver (1961); unless otherwise specified all quoted results refer to this hot extruded material. Owing to limitations of the extrusion method, considerable variations in oxygen and nitrogen content existed between extrusions and along the length of any one extrusion. The ranges of concentration for the common impurities are detailed in Table 1.

Other materials, which were investigated to a smaller extent, were:

- Pechiney powder direct extruded at 750 °C (Material B).
- Pechiney powder with 2 per cent. BeO added and direct extruded at 1050 °C (Material C).
- Pechiney powder oxidized at 800 °C in pure oxygen and then direct extruded at 1050 °C (Oxide content ≈ 2 per cent.) (Material D)
- Cast Pechiney powder extruded at 1050 °C (Material E).

The details of the materials are summarised in Table 1; a letter code has been allocated to the materials for reference throughout this report.

Specimens of four main types were used:

- Cylinders 0.4 inch dia. x 0.8 inch long or 0.3 inch dia. x 0.6 inch long for density, hardness, microscopy, and annealing studies.
- Cylinders 0.75 inch dia. x 3 inch long for neutron scattering and electrical resistivity measurements.

- Tensile test specimens with 2.1 inch length between shoulders and gauge diameters of 0.15 to 0.2 inch.

- Stored energy specimens ¾ inch dia. x 2 inch long with a central cavity.

All specimens were machined with the long dimension in the extrusion direction. To remove machining effects, specimens were first annealed at 800 °C for one hour in vacuum and air cooled. However this treatment did not result in sufficient reproducibility of tensile properties. To improve the reproducibility of results, an alternative treatment was used on later specimens (Rigs X75 and X84). This consisted of etching 0.005 inches of the surface of the specimens in a chromic–sulphuric–phosphoric acid solution before irradiation. All the tensile results quoted in this report were obtained with specimens treated in the latter way.

2.2 Irradiation Techniques

2.2.1 Pile temperature irradiations

In early irradiations at pile temperatures the specimens were held in carriers, packed in aluminium powder, and contained in an aluminium tube which was immersed directly into the heavy water. Thermocouples strapped to the specimens at intervals along the length of the rig indicated that the specimen temperatures were generally in the range 60 – 80 °C with some in one rig (X–4) reaching 120 °C. These irradiations were carried out in HIFAR in either the 2V–3 heavy water position or in the C–3 hollow fuel element position.

In later irradiations, the specimens were packed with aluminium powder and sealed in aluminium cans. These cans were loaded into and unloaded from a permanent irradiation facility (X–75) in the C–4 hollow fuel element position after retracting the facility into a handling flask situated on the storage block. No temperature measurement was made because sufficient experience had been gained with the earlier type of rig to guarantee that the irradiation temperatures would be in the range 60 – 100 °C.

2.2.2 High temperature irradiation

Irradiation at elevated temperatures was carried out in rigs such as that shown schematically in Figure 1. The specimens were supported in carriers in a stainless steel can which was filled with pure helium under reduced pressure. A Nichrome resistance furnace was wound on the outside of this can either on Refrasil insulation (Rig X–5) or on pyrophyllite ceramic formers. The furnace unit was contained in a splined aluminium former which allowed the passage of heater leads and thermocouples. Mineral–insulated thermocouples placed centrally in the top and bottom of each can were used for the measurement and control of temperature. In later rigs a third thermocouple placed eccentrically was included in each can to measure the radial temperature gradient in the can. Each rig contained three such furnace units in an outer aluminium tube which was immersed directly in the heavy water. The temperature of each furnace was controlled using a proportional potentiometer recorder–controller operating a magnetic amplifier and saturable reactor to control the heat input to each furnace unit. Temperature at the thermocouple position was maintained within ± 2 °C during pile operation but owing to uneven γ–heating effects, considerable longitudinal temperature gradients of up to 30 °C existed in each can. Radial temperature gradients were generally found to be less than 10 °C.

In the first rig of this type (X–5), the centre furnace unit failed during out–of–pile testing; nevertheless the irradiation was proceeded with and γ–heating alone was sufficient to raise the temperature of this unit to a useful operating temperature. It was found on disassembling the rig that the furnace had failed by short circuiting to the can; this made a hole in the can allowing severe oxidation of the specimens to occur.

In the second rig of this type, the centre furnace unit failed after one reactor operating period and the lower furnace unit after two periods. However, γ–heating was again sufficient to raise the temperatures to useful values during pile operation.

The temperature history of the specimen cans in these rigs is summarised in Table 2.

2.2.3 Flux monitoring

Integrated neutron fluxes were estimated from data on thermal/fast flux ratios obtained during the lower power operation of HIFAR, (Nicholson and Quealy 1961) and from cobalt monitors incorporated in each rig. The activity of the cobalt monitors after irradiation was compared with a standard monitor using a constant geometry γ-spectrometer; the standard monitor had been calibrated both by dissolution followed by $4\pi\beta$ counting, and with a Victoreen electrometer. For the short term irradiations the fast flux figures obtained by this method were checked by nickel monitors, the Co^{58} activity arising from the Ni^{58} (n,p) Co^{58} reaction being estimated using $\beta-\gamma$ coincidence counting methods.

The doses quoted in this report are the integrated fluxes over the fission neutron spectrum and the relative values for the hollow fuel element irradiations are probably accurate to $\pm 10-15$ per cent, but the absolute fluxes and the values for the 2V - 3 irradiations may have a larger error than this owing to uncertainties in the shape of the neutron energy spectrum. The fluxes above 1 MeV would be approximately 70 per cent. of the quoted integrated fission neutron fluxes.

Full details of the irradiation rigs, temperatures, doses, and specimens are given in Table 3.

2.3 Metrology and Density Measurement

Dimensions were measured to an accuracy of ±0.0001 inch using a vernier micrometer. Density was measured by the displacement method in n-octyl alcohol, the mean of two determinations being used. Reproducibility of the density changes to ±0.1 per cent. was obtained in earlier measurements and later with the use of a constant temperature room the reproducibility was improved to ±0.05 per cent.

2.4 Mechanical Testing

Tensile testing was carried out on an Instron 10,000 lb. machine using a crosshead speed of 0.002 in./minute. A linear differential transformer type extensometer with a 1 inch gauge length was used in the tests made at room temperatures but no extensometer was used in the elevated temperature tests; in these the extension was obtained from the crosshead movement and from measurements on the reassembled specimens after failure. In general only one specimen was tested for any one combination of conditions of dose, irradiation temperature, and testing temperature. Unirradiated material was tested in duplicate and excellent reproducibility was obtained.

Hardness tests were done on a standard Vickers machine using a 30 kg load. Three determinations were made on each specimen.

2.5 Determination of Gas Content

Helium and tritium contents were determined by the method of Hillen (1963 Unpublished). In this method the beryllium samples (0.2g) are melted in a vacuum fusion apparatus using a carbon crucible and R.F. heating. The gases given off are pumped away by a mercury diffusion pump into a Toepler pump where they are mixed with approximately 0.5 c.c. argon (STP) and the final gas volume measured. The collected gases are injected into a gas chromatograph which uses a 7ft. 6in., 1/8 inch int. dia. column of Linde 5A molecular sieve at 0°C with argon carrier gas. Helium and hydrogen present are detected with a thermistor detector (Perkin Elmer). Tritium is measured by passing the column effluent through a tube-type plastic scintillator detector coupled to a photo-multiplier tube. The chromatograph is calibrated with standard mixtures of hydrogen, helium and tritium in argon.

With 0.2g samples of beryllium metal it is possible to detect down to 10^{-2} c.c. He/g Be with an accuracy of ±5 per cent. The minimum limit of detection is approximately 5×10^{-4} c.c. He/g Be. The corresponding figures for tritium are approximately 100μ-curies and 5μ-curies T_2/g Be.

2.6 Stored Energy

Stored energy was measured with a differential calorimeter almost identical with that described by Clareborough, Hargreaves and West (1955). The irradiated specimen and an identical unirradiated specimen are located in an enclosure whose temperature is raised at a constant rate (2°/minute). By means of heaters inside the specimens their temperature is kept the same and identical with that of the enclosure. A sensitive differential wattmeter measures the difference in power to the two heaters and gives a direct measurement of any release of stored energy.

2.7 Long Wavelength Neutron Scattering

Long wavelength neutron scattering measurements were made on a spectrometer described by Sabine et al. (1962). The transmission of the irradiated and identical but unirradiated specimens is compared at various neutron wavelengths.

2.8 Out-of-Pile Controls

Control specimens for the elevated temperature irradiations were subjected to the same thermal history as the in-pile specimens. They were canned in stainless steel in an inert helium atmosphere for this heat treatment.

3. RESULTS

3.1 Density and Dimension Changes

No density or dimension changes could be detected in material irradiated at pile temperatures. Isothermal and isochronal post-irradiation annealing of specimens irradiated to various doses at pile temperatures were carried out. Decreases in density were observed to occur at temperatures of 800 °C and above in the irradiated material, but changes of the same order of magnitude were found to occur in unirradiated material at the same temperatures. The changes in unirradiated material varied widely from specimen to specimen and were probably due to the growth of fabrication pores. Owing to this effect the post-irradiation annealing results on density changes are of no significance.

Owing to surface oxidation, dimension changes could not be measured on specimens irradiated at elevated temperatures. However, after removing the oxidized surface layer by etching, density changes could be measured; these are summarised in Table 4 and the results for the high dose specimens are plotted in Figure 2. The changes in the control specimens were not significant. It can be seen that no significant changes occurred for temperatures of 500 – 560 °C but at 700 °C changes of up to 0.8 per cent. were observed. There appeared to be no significant difference between the different materials irradiated at the higher doses.

3.2 Gas Content

The helium and tritium content of various specimens is given in Table 5. Table 5 also gives calculated quantities of helium and tritium using the estimated fission fluxes and assuming a cross section of 100 mb for the (n, 2n) reaction and 25 mb for the (n, α) reaction. The agreement between the calculated and measured quantities for helium is generally very good considering the limitations in the dose measurement and the uncertainties in the cross section data. There are larger discrepancies (up to a factor of two) in the tritium contents.

3.3 Hardness Changes

The change in hardness with dose for material A irradiated at pile temperatures is plotted in Figure 3. There was no significant change until doses of above 10^{20} nvt were reached and thereafter the hardness increased in an approximately linear fashion with dose. The hardness changes in the specimens irradiated and heat treated at elevated temperatures are given in Table 4. Changes occurred in both the irradiated and heat treated specimens, particularly at the lower temperatures. Comparison of the irradiated and heat treated specimens showed that in general an increase in hardness occurred as a result of irradiation at 500 – 560 °C with a smaller change at 650 °C and little or no change at 680 – 700 °C although there were some exceptions to this pattern. It is interesting to note that the increase in hardness of material A after 1.4×10^{20} nvt at 500 °C was much greater than in the same material after 4×10^{20} nvt at 560 °C or after similar doses at 75 – 100 °C.

Hardness changes which occurred on isothermal and isochronal annealing of specimens irradiated at 75 – 100 °C are shown in Figures 4 – 7. The isochronal annealing results showed that recovery of the hardness-increase commenced at about 600 °C for one hour anneals and was virtually complete by 850 – 900 °C when changes in hardness of control specimens started to obscure the recovery process. In the isothermal anneals at 800 °C and 1000 °C recovery appeared to be complete in the first hour but at 600 °C changes continued to occur for periods up to 100 hours.

3.4 Tensile Properties

The results of tensile tests on specimens of material A irradiated to doses of between 5 x 10^{18} nvt and 3 x 10^{20} nvt at 75 – 100 °C (tested at temperatures in the range 100 – 600 °C) and of material B irradiated to 2 x 10^{20} nvt at 75 – 100 °C are given in Table 6. The results, plotted in Figures 8 – 11 and 12 – 13 for material A and material B respectively, show that there was little or no effect on any of the tensile properties for doses of 5 x 10^{19} and below. Above this dose there was an increase in proof stress and U.T.S. and a reduction in ductility, the change increasing with dose and decreasing with testing temperature. There was no marked difference in the changes in the hot extruded material A compared with the warm extruded material B.

The stress strain curves for all these specimens were smooth with no evidence of a yield point or multiple yielding. The shape of the stress–strain curves and the fracture appearance were not noticeably affected by irradiation.

In both the irradiated and unirradiated specimens the rate of work hardening following the elastic limit decreased with increasing temperature and above 400 °C very little work hardening occurred. Also the fracture appearance was similar in the irradiated and unirradiated material and followed the usual pattern, that is, a combination of cleavage and ductile shear failure at low temperatures with the proportion of ductile shear increasing with increasing temperature until at 400 °C the fracture was almost all by ductile shear with corresponding large reductions in area at the fracture. At higher temperatures there was very little reduction in area and the fracture became predominantly intergranular.

The results of tensile tests at room temperature on specimens irradiated to 1 – 1.5 x 10^{20} nvt at 500, 610, and 670 °C are given in Table 7 together with the tests on control specimens and on specimens that had been subjected to the same heat treatment. The shape of the curve showed considerable variations from one condition to another. The following points of interest were noted:

(i) Specimens tested in the as–received condition showed a smooth stress–strain curve with no evidence of a sharp yield point.

(ii) Specimens irradiated at the lower temperatures (500 °C) showed an increase in yield stress with a marked initial yield drop followed by multiple yielding to fracture. These specimens had a very low ductility.

(iii) In the case of irradiation at 610 and 670 °C, yield point effects were observed but the magnitude of the initial yield drop and the extent of multiple yielding was much lower. These specimens showed higher ductility than any of the control specimens.

(iv) All the heat–treated specimens showed an initial yield drop followed by multiple yielding over 0.4 – 0.8 per cent. extension. The yield point was in no instance as sharp as those of the specimens irradiated at 500 °C and the extent of the discontinuities in the stress–strain curve appeared to increase with temperature rather than decrease as in the irradiated specimens.

The results of tensile tests on material A irradiated (in Rig X–84) at elevated temperatures to doses of about 2 x 10^{20} nvt and tested over the range from room temperature to 600 °C are given in Table 8 and are plotted in Figures 14 – 17. Tests of as–received specimens and those subjected to the same heat treatment as the irradiated specimens are also shown. These tests were made to elucidate further the yield point effects and low room temperature ductility observed in specimens irradiated to 1.4 x 10^{20} nvt at about 500 °C (see last paragraph). The specimens in can A in this rig operated in the range 480 – 510 °C during the irradiation and those in can B in the range 580 – 610 °C. However, owing to a furnace failure and uneven gamma heating effects those in can C operated for approximately the first 60 per cent. of the irradiation at 480 – 550 °C and for the remaining period at 300 – 400 °C.

Comparison of the heat treated material with the as–received material shows that marked property changes occurred as a result of heat treatment alone; increases in U.T.S., proof stress, and ductility were observed at all testing and heat treatment temperatures.

Comparison of the irradiated specimens with the heat treated specimens shows the effects of irradiation over and above heat treatment. The following features were observed:

(i) The proof stress increased as a result of irradiation at all testing temperatures, the change being greatest for specimens from can C tested at low temperatures and becoming very small for all materials at the higher temperatures.

(ii) The ductility of specimens from can B, that is, the highest temperature can, was not unduly affected at any testing temperature. However, the low temperature ductility of specimens from the lower temperature cans A and C was reduced considerably, the ductility being zero at room temperature and only half of that of the control specimens at 100 and 200 °C. For temperatures of 300 °C and above however, the changes in ductility were very small.

(iii) The reduced ductility of these specimens was reflected in lower U.T.S. values at the lower testing temperatures but the changes in U.T.S. of all specimens at testing temperature of 300 °C and above was very small.

Yield point effects were observed in all the irradiated specimens at testing temperatures of up to 400 °C but were not seen in any of the heat treated or irradiated specimens at testing temperatures above 500 °C. The specimens from can A and can B (irradiated at 480 – 510 °C and 580 – 610 °C) and tested up to 300 °C showed little or no yield drop but a region of easy glide up to extensions of 0.6 per cent. before work hardening commenced. At 400 °C a yield point drop was observed but little or no easy glide. The specimens from can C irradiated at lower temperatures showed a very marked yield drop when tested at room temperature and 200 °C; at 200 °C and 300 °C multiple yielding, similar to that observed in specimens from Rig X–5, occurred up to extensions of 3 per cent. The specimen from can C tested at 400 °C showed a broad yield point effect in that after yielding at 29,600 p.s.i. the stress increased to 31,600 p.s.i. over an extension of about 0.16 per cent. and then decreased to the original level at the same rate; the extension then continued in a normal manner. A similar but less pronounced effect was observed in the test at 500 °C. The mode of fracture did not appear to be altered significantly by irradiation and followed the usual pattern with testing temperature as described earlier.

3.5 Metallography

Metallographic examination failed to show evidence in any specimens for void formation during irradiation.

Longitudinal sections of the gauge lengths of tensile specimens were examined and the following observations made:

(i) No significant differences could be observed between irradiated and unirradiated specimens for any condition.

(ii) The fracture face appearance was in accordance with the macroscopic observations reported in the last section, that is, intracrystalline failure by cleavage and ductile shear at low temperatures changing over to intercrystalline failure at elevated temperatures.

(iii) The intercrystalline failure at elevated temperatures was generally associated with void formation and extensive transverse cracking by link–up of voids near the fracture surface.

3.6 Electron Microscopy

Replicas taken from surfaces of specimens fractured at room temperature were examined by electron microscopy. Full details of these investigations have been published (Chute 1963). The most important observations and conclusions were:

(i) Voids assumed to be helium bubbles were observed in grain boundaries of material irradiated to high doses at elevated temperatures or irradiated at pile temperatures and annealed at elevated temperatures.

(ii) The conditions at which bubbles were first observed and the subsequent size and number of bubbles depended to a very marked extent on the material history as well as on the dose and temperature. These variations occurred not only between material fabricated by different routes but also between materials fabricated by the same route but subjected to different heat treatments before irradiation.

(iii) The hot extruded material A which had been heat treated before irradiation showed a smaller amount of bubble formation than either the same material without prior heat treatment or materials B and E. No bubbles were observed in material A at irradiation temperatures of up to 680 °C after 1.5 x 10^{20} nvt or after 4 x 10^{20} nvt at 560 °C.

(iv) Addition of beryllium oxide either by pre-oxidizing the powder (material C) or by additions of BeO before extrusion did not appear to affect the bubble distribution in hot extruded material.

(v) The hot extruded material with prior heat treatment showed a much smaller amount of bubble formation than any of the materials fabricated in the U.K. and subjected to the same irradiation history (Hickman et al. 1962).

(vi) The only material in which significant bubble formation on cleavage surfaces was observed was the coarse grained material E.

(vii) Little direct evidence was found for nucleation of bubbles at second phase precipitates, but it was concluded from this work that this was the only reasonable explanation of the wide range of behaviour between different materials. Limitations in the replica technique precluded any firm confirmation of this point. (The minimum size of bubble which could be observed was about 150 Å and it is possible that all the helium could be contained in smaller bubbles).

(viii) It was definitely established that at high temperatures large bubbles grow at the expense of smaller ones, the only acceptable explanation being that re-solution of helium occurs.

3.7 Electrical Resistivity

The electrical resistivity changes in irradiated material A were studied and the annealing of these changes observed. The experiments will be described in detail by Svenson and Hickman (1963). The main observations and conclusions were:

(i) The resistivity increased in a perfectly linear manner with dose up to the highest dose measured (6 x 10^{20} nvt). It was concluded that this resistivity change could only be due to helium in enforced solid solution and it was estimated that the change was of the order of 12 μ ohm-cm per 1 per cent. of helium.

(ii) The resistivity increase recovered in a steady manner over the range 300 - 900 °C in material annealed before irradiation. Analysis of the isothermal and the isochronal annealing data showed that the recovery was not a uniquely activated process but occurred over a wide spectrum of activation energies. However material which was not annealed before irradiation did not start recovering until 500 °C and recovery was complete at 800 °C.

(iii) These results were interpreted in terms of differing scales of bubble nucleation in the two materials. In the material annealed before irradiation, bubble nucleation was thought to occur on a very fine scale and because diffusion had only short distances over which to occur, recovery commenced at low temperatures. In the second material recovery did not start until higher temperatures were reached because nucleation occurred on a coarser scale.

3.8 Long Wavelength Neutron Scattering

Long wavelength neutron scattering measurements have been used successfully for measuring defect concentrations in irradiated beryllium oxide and other materials (Sabine et al. 1962). The method depends on the fact that neutrons are scattered by defects. In materials of low capture cross section and with neutrons whose wavelength is beyond the Bragg cut-off when coherent elastic scattering is zero, the scattering from defects can form a significant part of the total cross section.

Measurements were made on specimens of material A irradiated to 6 x 10^{20} nvt but no change in cross section could be observed (T.M. Sabine, A.A.E.C. unpublished). This places an upper limit of about 0.05 per cent. on the defect concentration in the irradiated material.

3.9 Stored Energy

Stored energy measurements showed no detectable release up to 500 °C. The limit of detection would be about 0.05 cal/g. Above 500 °C it was not possible to obtain reliable measurements owing to interfering effects from precipitation reactions.

4. DISCUSSION

4.1 Swelling

The observed density changes were generally small, the largest being 0.8 per cent. in material irradiated to 6×10^{20} nvt at 700 °C. A change of 0.8 per cent. means that the pressure in the gas bubbles was of the order of 200 atmospheres. For the gas pressure to be in equilibrium with the surface tension the mean bubble size must have been about 0.1μ which is of the same order as that observed by electron microscopy.

The density changes did not appear to vary significantly with material although the electron microscopy showed considerable variations between materials. However the accuracy of the density measurements was low and the volume increase observed in unirradiated material, due to growth of pre-existing pores, complicates the interpretation.

4.2 Mechanical Properties

After irradiation at 100 °C little or no change in mechanical properties or electrical resistivity was observed until the dose exceeded about 10^{20} nvt; this suggests that defect production was not important and all defects had annealed out by this temperature. In other metals where defects remain at these temperatures, large changes in mechanical properties and resistivity are observed at doses of $10^{18} - 10^{19}$ nvt and the changes are often approaching saturation by 10^{20} nvt. The above interpretation is supported by Blewitt's (1958) observation that all the damage in beryllium irradiated at 20 °K, as measured by changes in resistivity, had annealed out by 300 °K. The absence of any detectable long wavelength neutron scattering effect (Section 3.8) also shows that no defects were present after the 100 °C irradiation.

The observed changes in mechanical properties can be satisfactorily interpreted, qualitatively at least, in terms of the helium distribution.

The changes in mechanical properties which were observed were of three main types depending on the irradiation temperature:

(i) After irradiation at 75 – 100 °C there was an increase in hardness, and yield strength and a reduction in ductility for low testing temperatures. At higher testing temperatures there was still an increase in yield strength but the changes in ductility became much smaller as the testing temperature increased. Materials A and B behaved in a similar manner under these conditions.

(ii) For irradiation at about 400 – 500 °C material A showed very large increases in yield strength and decreases in ductility at low testing temperatures; the changes were much greater than for the equivalent doses at 75 – 100 °C and were associated with the development of yield point effects. With increasing testing temperature both the ductility and the yield strength changes became smaller, being negligible in the case of ductility by 300 °C and, in the case of yield strength, by 500 °C. These effects were not observed in materials fabricated in the U.K., after similar irradiation conditions (Hickman et al. 1962).

(iii) For irradiation at above 600 °C there was little or no change in the properties at any testing temperatures of the hot extruded material A, although previous work on other materials fabricated in the U.K. (Hickman et al. 1962) had shown that a large decrease in ductility occurred for these conditions.

The changes in properties at low temperatures were apparently a result of solid solution type hardening by the helium produced under irradiation. The changes were somewhat larger than one would normally expect with this concentration of solute unless segregation occurred, but this was probably due to the fact that the helium was in enforced solid solution and the strain around the solute atoms

would be large. Although the Goldschmidt radius for beryllium (1.22 Å) is not very different from the atomic radius of helium (1.13 Å), electronic effects would probably result in a large strain field. The relatively small temperature dependence of the change in yield strength and the absence of yield point effects supports the suggestion that the changes were due to simple solid solution hardening. As would be expected from simple solid solution hardening, the changes in mechanical properties did not appear to vary significantly with material history. The property measurements at temperatures above 300 °C on material irradiated at 75 – 100 °C should be interpreted with some caution as the helium could have been mobile at these temperatures and changes in helium distribution may have occurred during the test.

As the irradiation temperature was raised the helium became mobile and was precipitated, and the observed changes in mechanical properties in material A were very similar to those which occur in precipitation hardening alloys. At intermediate temperatures the helium bubbles were evidently dispersed on a very fine scale and this caused the large increases in low temperature yield strength and the associated very low ductility.

Barnes (1961) has stated that because little or no strain field is observed around bubbles in thin film electron microscopy studies, the interaction energy between a dislocation and a bubble is simply due to the elimination of the length of the dislocation which passes through the bubble. He calculates that the stress necessary to force the dislocation line through the bubbles is given by:

$$\sigma = Gb\,(2nr)^{\frac{1}{2}},$$

where G = shear modulus

b = Burgess vector

n = number of bubbles/cm^3

r = radius of bubbles.

Assuming that the helium is in equilibrium with the surface tension, this stress will exceed the flow stress when the bubbles are about 2×10^{-7} cm in diameter and the bubble density is about 5×10^{15} cm^3 for specimens irradiated to about 2×10^{20} nvt at 500 °C.

This bubble size is not incompatible with that deduced from the electrical resistivity measurements (Svenson and Hickman 1963), but is too small for observation by electron microscopy. Barnes' assumption that there is no strain field around the bubbles may not hold down to very small bubble sizes but nevertheless the above analysis shows that it is quite possible for bubbles at a suitable distribution to produce the observed effects.

The strong temperature dependence of the change in yield strength under these conditions and the observations of yield point effects and easy glide regions suggest that a lot of the change was due to source hardening by precipitation of bubbles at dislocations but the wide variety of effects observed for very similar conditions and the complicating effects of heat treatment preclude any quantitative analysis of the data in terms of dislocation theory.

The electrical resistivity measurements indicated that the helium became mobile in this material at temperatures as low as 300 °C and the fact that the hardening was greater in specimens (from Rig X–84) which had been at 300 – 400 °C for a large part of the time than in specimens which were irradiated for the whole period at 500 °C suggests that this initial hardening stage may be important down to 300 – 400 °C. Further experiments are needed to check this point. These effects were evidently not observed in the U.K. fabricated materials because bubble nucleation did not occur on a fine scale within the grains owing to lack of suitable nuclei.

At irradiation temperatures above 500 °C the bubbles became coarser and an "overaged" condition was reached in which there was little or no change in the properties at low temperatures. However, at 600 °C a significant proportion of the helium formed bubbles at the grain boundaries and it has been shown previously that this can cause a serious loss in high temperature ductility (Hickman et al. 1962). The hot extruded material A however, did not show any significant loss in high temperature properties under these conditions at least at the highest dose investigated.

The electron microscopy work showed that there was much smaller precipitation of bubbles at the grain boundaries in this material than in the materials fabricated in the U.K. which showed the serious loss in high temperature ductility. The loss in ductility in the U.K. material was attributed to the helium bubbles acting as nuclei for the formation of grain boundary voids under stress which then resulted in premature intergranular failure.

The smaller size and number of bubbles in the material A used in this investigation has resulted in the smaller changes in high temperature properties. As was mentioned earlier, heat treatment of this material before irradiation resulted in an even smaller amount of helium precipitation at the grain boundaries. The specimens (from Rig X-84) which showed little change in high temperature properties were not heat treated before irradiation so heat treatment should improve the situation even further.

It is also important to note that compared with material A the material C prepared from pre-oxidized powder showed little difference in behaviour as far as the helium precipitation was concerned. This material is important as it has excellent corrosion resistance (Smith et al. 1961). The high temperature mechanical properties in the unirradiated condition were not very different from the normal material and one would not therefore expect the mechanical properties of the irradiated material to be very different from the results reported for the material A.

4.3 Mechanism of Nucleation and Growth of Gas Bubbles

The experiments described in this report do not provide much further information on the mechanism of nucleation and growth of bubbles beyond that discussed in the reports on the electron microscopy (Chute 1963) and electrical resistivity investigations (Svenson and Hickman 1963). The mechanical property work confirmed that as the temperature is raised the number of bubbles decreases and their size increases, that is, that the larger bubbles grow at the expense of the smaller bubbles. As was discussed by Chute (1963), the only explanation that can be offered of this phenomenon is that re-solution of helium must occur and that at least at elevated temperatures there must be a small solubility of helium in beryllium.

The work has also emphasised the importance of material history in determining the scale of bubble nucleation. The mechanical property work showed that nucleation of bubbles must have occurred within the grains in some materials as well as at grain boundaries although these never grew to a size sufficient to be observed by electron microscopy, at least in material A.

4.4 Future Work

It is unfortunate that the importance of material history was not fully realised when these investigations were planned. The difficulty in controlling the content and distribution of impurities in beryllium together with the curtailment of the beryllium programme at Lucas Heights means that the work will not be taken through to its logical conclusion. However some additional work is still in progress. This is mainly a continuation of the mechanical property studies to higher doses on both the hot extruded material and the hot extruded pre-oxidized material in order to confirm the superior high temperature properties of this material. The effect of vacuum hot pressing before extrusion and of pre-irradiation heat treatment are being investigated further and a limited investigation of bubble nucleation and growth using thin film electron microscopy is also planned.

5. CONCLUSIONS

The property changes in beryllium metal fabricated by various routes have been investigated after irradiation to fission neutron doses up to 6×10^{20} nvt at temperatures from 75 °C to 700 °C. The following conclusions were reached:

(i) Displacement damage is not significant in beryllium metal at irradiation temperatures of 75 °C and above. All the observed property changes can be interpreted in terms of the distribution of helium produced by nuclear reactions in the material.

(ii) Swelling in beryllium due to helium bubble formation is not a serious technological problem at doses approaching 10^{21} nvt at least for temperatures of 700 °C and below. The maximum observed swelling was 0.8 per cent. after a dose of 6×10^{20} nvt at 700 °C.

(iii) In irradiation of beryllium at temperatures of about 75 – 100 °C solid solution hardening occurs due to the helium being in enforced solid solution in the lattice. This results in increases in yield strength and decreases in ductility at low testing temperatures but the material retains substantial ductility in the extrusion direction. These changes in properties do not depend to any marked extent on the material history.

(iv) After irradiation of the hot extruded material A to 2 x 10^{20} nvt at temperatures of about 500 °C there was a very marked reduction in ductility at low temperatures. Specimens showed zero ductility in the extrusion direction at room temperature. This effect was associated with large increases in yield strength and the appearance of yield point effects in the stress–strain curve; it is attributed to the early stages of nucleation and growth of gas bubbles. At testing temperatures of 300 °C and above the effect disappears. This effect may be important for irradiation temperatures down to 300 °C but was not observed in some materials in which bubble nucleation does not occur to any extent within the grains owing to lack of suitable nuclei.

(v) For irradiation temperatures of 600 °C and above there was little or no change in the properties of Lucas Heights hot extruded material at any temperature, at least up to doses of 2 x 10^{20} nvt. This is attributed to coarsening of the bubbles beyond the critical stage for hardening. The properties of this hot extruded Pechiney material under these conditions (that is after irradiation at 600 °C and above) were superior to those of material fabricated in the U.K. which showed serious reduction of high temperature ductility under similar conditions. This superiority is thought to be due to the fact that the amount of bubble precipitation on grain boundaries was much less than for the U.K. fabricated material because the majority of the helium had precipitated on a fine scale within the grains.

(vi) A pre–irradiation heat treatment consisting of annealing for one hour at 800 °C and air cooling further reduced the grain boundary bubble precipitation.

(vii) Addition of oxide to the beryllium before fabrication did not significantly affect the bubble distribution and therefore would not be expected to affect the mechanical property changes on irradiation.

(viii) Although no direct evidence was obtained, the only satisfactory explanation of the difference in behaviour under irradiation of materials prepared by different routes is that the bubble nucleation occurred at a second phase, and the pre–irradiation history significantly affects the distribution of this second phase.

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TABLE 1

DETAILS OF MATERIALS USED IN THE INVESTIGATION

Material Code	Fabrication Method	Extrusion Temperature	Reduction Ratio	Density	Typical Analysis					
					Fe p.p.m.	Al p.p.m.	Si p.p.m.	N p.p.m.	Cl p.p.m.	O %
A	Hot extruded	1050 °C	20 : 1 to 44 : 1	1.82 – 1.84	240–600	100–500	100–400	110–3300	< 100	0.4 – 0.7
B	Warm extruded	750 °C	11 : 1 to 16 : 1	1.83 – 1.95	1300	1250	560	300	n.d.	0.7
C	2% BeO powder added then hot extruded	1000 °C	16 : 1	1.84 – 1.86						
D	Hot extruded from pre–oxidized powder	1000 °C	16 : 1	1.85 – 1.87						
E	Vacuum cast and hot extruded	1000 °C	16 : 1	1.84 – 1.85						

TABLE 2

OPERATING TEMPERATURE HISTORY OF HIGH TEMPERATURE RIGS

The usual operating temperature range is that in which the specimens spent at least 95 per cent. of their irradiation time. The mean operating temperatures are rough estimates which have been used in the text for easy reference.

Rig No.	Can No.	Max. Temperature °C	Min. Temperature °C	Usual Operating Range °C	Mean Operating Temperature °C
X – 5	A	530	450	480 – 510	500
	B	640	520	570 – 630	600
	C	710	600	650 – 700	670
X – 73	A	610	520	550 – 570	560
	B	840	670	680 – 730	700
	C	740	630	640 – 680	660
X – 84	A	510	460	480 – 510	500
	B	580	610	580 – 610	600
	C	540	310	480 – 540 for 45 days	–
				300 – 350 for 35 days	

TABLE 3

DETAILS OF SPECIMENS, IRRADIATION RIGS, AND IRRADIATION CONDITIONS

Rig No.	Irradiation Position	Material	No. of Specimens	Specimen Size inches	Purpose	Nominal Irradiation Temperature °C	Irradiation Dose nvt	Pre–irradiation Treatment
X–22	2V–3	A	2	3/4" ϕ x 3"	Resistivity and neutron scattering	75 – 100	2.5×10^{19}	Annealed for one hour at 800 °C and air cooled
		A	8	0.3" ϕ x 0.6"	Density, annealing studies	"	2×10^{19}	" " " "
X–38	"	A	2	3/4" ϕ x 3"	Resistivity and neutron scattering	"	5.8×10^{19}	" " " "
			8	0.3" ϕ x 0.6"	Density, annealing studies	"	3.5×10^{19}	" " " "
X–4	C–3 H.F.E.	A	2	3/4" ϕ x 3"	Resistivity and neutron scattering	"	2.0×10^{20}	" " " "
		A	8	0.3" ϕ x 0.6"	Density, hardness, annealing studies	"	1.3×10^{20}	" " " "
X–39	"	A	2	3/4" ϕ x 3"	Resistivity and neutron scattering		6.0×10^{20}	" " " "
		A	8	0.3" ϕ x 0.6"	Density, hardness, annealing studies	"	5.0×10^{20}	" " " "
X–75	C–2 H.F.E.	A	6	Standard Tensile	Tensile testing	"	6×10^{18}	Etched in chromic–phosphoric–sulphuric mixture
		A	6	"	"	"	1×10^{19}	" " " "
		A	6	"	"	"	5×10^{19}	" " " "
		A	6	"	"	"	2×10^{20}	" " " "
		A	6	"	"	"	3×10^{20}	" " " "
		B	6	"	"	"	2×10^{20}	" " " "

X–5	C–1 H.F.E.	A	4	"	"	600	9×10^{19}	Annealed for one hour at 800 °C and air cooled
		A	4	"	"	670	1.5×10^{20}	" " " "
		A	4	"	"	500	1.4×10^{20}	" " " "
		A	4	0.3" ϕ x 0.6"	General	600	9×10^{19}	" " " "
		A	4	"	"	670	1.5×10^{20}	" " " "
		A	4	"	"	500	1.4×10^{20}	" " " "
X–84	C–5 H.F.E.	A	8	Standard Tensile	Tensile testing	500	1.5×10^{20}	Etched in chromic–sulphuric–phosphoric mixture
			8	"	"	600	2.5×10^{20}	" " " "
			8	"	"	300 – 500	2.4×10^{20}	" " " "
X–73	C–1 H.F.E.	A	4	0.3" ϕ x 0.6"	General	560	4.0×10^{20}	Annealed for one hour at 800 °C and air cooled
		A	4	"	"	700	6.0×10^{20}	" " " "
		A	4	"	"	660	5.5×10^{20}	" " " "
		C	4	"	"	560	4.0×10^{20}	" " " "
		C	4	"	"	700	6.0×10^{20}	" " " "
		C	4	"	"	660	5.5×10^{20}	" " " "
		D	4	"	"	560	4.0×10^{20}	" " " "
		D	4	"	"	700	6.0×10^{20}	" " " "
		D	4	"	"	660	5.5×10^{20}	" " " "
		E	2	"	"	560	4.0×10^{20}	" " " "
		E	2	"	"	700	6.0×10^{20}	" " " "
		E	2	"	"	660	5.5×10^{20}	" " " "

TABLE 4

DENSITY AND HARDNESS CHANGES AFTER IRRADIATION TO VARIOUS DOSES AT ELEVATED TEMPERATURES

Material	Irradiation or Heat Treatment Temp. °C	Irradiation Dose nvt	Density Change %	Hardness Change DPN
A	500	1.4×10^{20}	−0.15	+ 15 − 25
	600	0.9×10^{20}	−0.2	< 5
	670	1.5×10^{20}	−0.4	< 5
	560	4.0×10^{20}	−0.22	+ 1
	”	Control	0	− 3
	650	5.5×10^{20}	−0.49	− 1
	”	Control	−0.05	− 5
	700	6.0×10^{20}	−0.82	0
	”	Control	+0.05	− 1
C	560	4.0×10^{20}	+0.05	+14
	”	Control	+0.05	+1
	660	5.5×10^{20}	−0.27	+3
	”	Control	+0.05	+7
	700	6.0×10^{20}	−0.70	− 1
	”	Control	−0.16	− 1
D	560	4.0×10^{20}	0	+4
	”	Control	−0.11	− 3
	650	5.5×10^{20}	−0.54	0
	”	Control	−0.16	− 5
	700	6.0×10^{20}	−0.80	− 7
	”	Control	−0.11	− 9
E	560	4.0×10^{20}	0	+11
	”	Control	+0.27	− 4
	660	5.5×10^{20}	−0.16	+ 7
	”	Control	+0.16	+ 6
	700	6.0×10^{20}	−0.60	+11
	”	Control	+0.11	− 1

TABLE 5

COMPARISON OF MEASURED AND CALCULATED QUANTITIES OF HELIUM AND TRITIUM

Rig No.	Irradiation Temperature °C	Helium Content		Tritium Content	
		Measured c.c./g	Calculated c.c./g	Measured mc/g	Calculated m.c/g
X–75	75 – 100	7×10^{-3}	9×10^{-3}	0.019	0.075
		0.146	0.15	6.2	9.0
		0.168	0.175	8.4	10.2
		0.134	0.14	6.1	8.4
		0.103	0.11	4.7	6.6
X–39	75 – 100	0.360	0.32	–	–
X–84	500	0.069	0.09	1.4	5.4
	600	0.146	0.15	5.6	9.0
	300 – 500	0.121	0.14	4.4	8.8
X–73	560	0.22	0.25	–	–
	700	0.334	0.36	21.2	21.0
	660	0.271	0.30	–	–

TABLE 6

RESULTS OF TENSILE TESTS ON MATERIAL IRRADIATED AT 75 – 100°C TO VARIOUS DOSES

Material	Dose nvt	Test Temperature °C	U.T.S. p.s.i.	0.1% P.S. p.s.i.	Elongation %	R. of A. %	Fracture
A	Control	R.T.	70,100	32,400	20.0	20.5	Cleavage (90%) and ductile shear
,,	,,	,,	–	33,900	–	–	Cleavage (100%)
,,	,,	100	55,000	29,100	17.6	18.1	Cleavage (75%) and shear
,,	,,	,,	62,200	34,400	19.7	21.8	Cleavage (90%) and shear
,,	,,	200	40,500	23,900	22.7	28.6	Cleavage (40%) and shear
,,	,,	,,	42,000	22,000	22.8	26.9	Cleavage (40%) and shear
,,	,,	300	32,200	18,600	20.0	30.8	Cleavage (5%) and shear
,,	,,	,,	32,000	17,600	22.7	31.6	Cleavage (30%) and shear
,,	,,	400	23,100	15,600	11.9	23.3	Shear
,,	,,	,,	25,700	16,300	13.2	27.4	Shear
,,	,,	500	12,500	11,400	5.9	4.7	Intergranular
,,	,,	,,	15,000	12,500	8.1	8.2	Intergranular
,,	,,	600	4,600	4,200	4.9	3.0	Intergranular
,,	,,	,,	4,700	4,400	5.2	3.3	Intergranular
,,	,,	700	1,500	1,300	10.1	5.2	Intergranular
,,	,,	,,	1,500	1,400	9.0	5.1	Intergranular
,,	5×10^{18}	100	55,300	30,200	21.3	21.5	Cleavage (80%) and shear
,,	,,	200	42,900	24,300	24.2	27.5	Cleavage (40%) and shear
,,	,,	300	35,300	22,600	18.8	22.6	Cleavage (15%) and shear
,,	,,	400	26,100	17,800	10.0	25.7	Shear
,,	,,	500	15,000	12,800	6.4	5.4	Intergranular
,,	,,	600	4,600	4,500	5.4	4.9	Intergranular
,,	1×10^{19}	100	54,300	24,600	19.6	22.5	Cleavage (70%) and shear
,,	,,	200	41,700	24,000	20.7	29.2	Cleavage (30%) and shear
,,	,,	300	34,500	22,700	13.9	32.4	Cleavage (5%) and shear
,,	,,	400	25,600	19,200	9.0	25.1	Shear
,,	,,	500	15,400	14,200	6.3	6.1	Intergranular
,,	,,	600	4,600	4,200	6.0	5.1	Intergranular
,,	5×10^{19}	100	61,100	31,300	18.8	22.6	Cleavage (80%) and shear
,,	,,	200	44,700	26,900	14.6	26.4	Cleavage (25%) and shear
,,	,,	300	34,800	21,500	11.5	29.1	Cleavage (20%) and shear

(continued)

TABLE 6 (continued)

Material	Dose nvt	Test Temperature °C	U.T.S. p.s.i.	0.1% P.S. p.s.i.	Elongation %	R. of A. %	Fracture
A	5×10^{19}	400	25,700	17,700	8.3	22.7	Cleavage (2%) and shear
,,	,,	500	15,100	14,200	4.9	6.5	Intergranular
,,	,,	600	4,400	4,400	4.9	4.2	Intergranular
,,	2×10^{20}	100	65,200	40,800	9.9	18.4	Cleavage (75%) and shear
,,	,,	200	51,800	32,800	11.4	20.4	Cleavage (40%) and shear
,,	,,	300	44,300	28,100	9.9	19.1	Cleavage (85%) and shear
,,	,,	400	36,500	26,100	7.7	16.8	Cleavage (5%) and shear
,,	,,	500	21,500	19,300	3.2	1.7	Cleavage (20%) and intergranular
,,	3×10^{20}	100	79,700	48,800	11.0	19.2	Cleavage (90%) and shear
,,	,,	200	62,000	39,800	12.0	21.2	Cleavage (70%) and shear
,,	,,	300	51,200	33,700	8.4	23.4	Cleavage (50%) and shear
,,	,,	400	39,300	30,200	7.1	18.3	Cleavage (20%) and shear
,,	,,	500	23,000	22,100	4.8	0.5	Cleavage (50%) and intergranular
,,	,,	600	5,500	5,100	4.1	7.1	Intergranular
B	Control	100	88,200	58,600	16.9	17.9	Cleavage (90%) and shear
,,	,,	200	70,100	53,800	20.2	22.4	Cleavage (75%) and shear
,,	,,	300	49,000	43,600	15.1	22.4	Cleavage (10%) and shear
,,	,,	400	31,300	30,400	9.9	20.2	Shear
,,	,,	500	12,100	11,500	9.4	9.0	Intergranular
,,	,,	600	6,800	6,200	5.4	2.1	Intergranular
,,	2×10^{20}	100	89,300	63,000	5.5	9.3	Cleavage (95%) and shear
,,	,,	200	–	55,000	–	–	Cleavage
,,	,,	300	58,300	47,500	8.8	19.1	Cleavage (30%) and shear
,,	,,	400	44,500	38,200	8.7	13.3	Shear
,,	,,	500	20,600	19,700	6.5	4.8	Intergranular
,,	,,	600	7,300	7,000	4.9	1.7	Intergranular

TABLE 7

RESULTS OF TENSILE TESTS AT ROOM TEMPERATURE ON MATERIAL A IRRADIATED TO VARIOUS DOSES AT ELEVATED TEMPERATURES IN RIG X-5

Rig No.	Dose nvt	Irradiation or Heat Treatment Temp. °C	Testing Temperature °C	U.T.S. p.s.i.	0.1% P.S. p.s.i.	Yield Stress p.s.i.	Elongation %	R. of A. %	Fracture	Multiple Yielding %
X5 Can A	8.5×10^{19}	600	R.T.	66,000		51,300	1.8	1.6	Cleavage	0.6
"	"	"	"	83,900		Upper 50,900 Lower 50,300	11.3	11	Cleavage	0.6
"	"	"	"	85,700		51,500	9.5	8.6	Cleavage	0.7
"	"	"	"	86,900		50,000	11.7	n.d.	Cleavage	0.6
"	Control	"	"	96,200		Upper 61,600 Lower 61,500	3.9	n.d.	Cleavage	0.4
X5 Can B	1.5×10^{20}	670	"	82,300	47,600		9.0	n.d.	Cleavage	
"	"	"	"	78,800	48,600		7.9	8.1	Cleavage	
"	"	"	"	84,000		47,900	9.3	n.d.	Cleavage	0.5
"	Control	"	"	81,200		Upper 54,200 Lower 53,300	4.8	5.6	Cleavage	0.8
"	Control	"	"	79,300		Upper 50,100 Lower 50,000	5.8	n.d.	Cleavage	0.6
X5 Can C	1.4×10^{20}	500	"	53,800		Upper 52,600 Lower 51,600	1.0	n.d.	Cleavage	1.0
"	"	"	"	58,700		Upper 61,000 Lower 56,900	1.2	0.6	Cleavage	1.2
"	"	"	"	60,800		Upper 62,600 Lower 60,500	0.4	0.5	Cleavage	0.4
"	Control	"	"	69,200		46,700	2.7	1.1	Cleavage	

RESULTS OF TENSILE TESTS AT VARIOUS TEMPERATURES ON MATERIAL A IRRADIATED TO 1.5 TO 2.5 x 10^{20} nvt AT ELEVATED TEMPERATURES

Rig No.	Dose nvt	Irradiation or Heat Treatment Temp. °C	Testing Temperature °C	U.T.S. p.s.i.	0.1% P.S. p.s.i.	Yield Stress p.s.i.	Elongation %	R. of A. %	Fracture	% Extension over which Multiple Yielding is Observed
X84 Can A	1.5 x 10^{20}	500	R.T.	58,300		52,600	0.9	1.0	Cleavage	0.28
,,	,,	,,	100	74,600		Upper 52,900 Lower 50,900	12.9	12.0	95% Cleavage & Ductile Shear	0.58
,,	,,	,,	200	55,900		43,100	21.1	33.0	95% Cleavage & Ductile Shear	0.5
,,	,,	,,	300	41,800		37,600	36.8	44.4	40% Cleavage & Ductile Shear	0.63
,,	,,	,,	400	28,400		Upper 28,200 Lower 27,200	19.3	40.7	5% Cleavage & Ductile Shear	–
,,	,,	,,	500	20,500	19,500		9.1	10.5	Intergranular	–
,,	,,	,,	600	9,700	9,400		5.3	3.9	Intergranular	–
,,	Control	,,	R.T.	91,500	42,500		19.1	18.4	95% Cleavage & Ductile Shear	–
,,	,,	,,	200	52,600	34,100		34.3	47.5	90% Cleavage & Ductile Shear	–
,,	,,	,,	300	37,200	27,900		28.7	49.5	20% Cleavage & Ductile Shear	–
,,	,,	,,	400	27,000	23,100		22.2	41.5	Ductile Shear	–
,,	,,	,,	600	8,200	8,100		7.3	6.4	Intergranular	–
X84 Can B	2.5 x 10^{20}	600	R.T.	88,600		Upper 54,000 Lower 53,500	13.6	12.5	95% Cleavage & Ductile Shear	–
,,	,,	,,	100	77,500		50,300	23.1	19.6	95% Cleavage & Ductile Shear	0.34
,,	2.4 x 10^{20}	,,	200	56,100		Upper 41,500 Lower 39,800	36.6	37.2	92% Cleavage & Ductile Shear	0.48
,,	,,	,,	300	40,600		33,900	31.0	43.6	50% Cleavage & Ductile Shear	0.67

(continued)

TABLE 8 (continued)

Rig No.	Dose nvt	Irradiation or Heat Treatment °C	Testing Temperature °C	U.T.S. p.s.i.	0.1% P.S. p.s.i.	Yield Stress p.s.i.	Elongation %	R. of A. %	Fracture	% Extension over which Multiple Yielding is Observed
X84 Can B	2.4×10^{20}	600	400	31,500		Upper 29,900 Lower 29,500	26.0	32.7	Ductile Shear	0.09
"	2.5×10^{20}	"	500	17,700	17,500		9.7	15.1	Intergranular	–
"	"	"	600	10,300	9,900		6.3	6.1	Intergranular	–
"	Control	"	R.T.	91,700	46,600		18.9	17.4	95% Cleavage & Ductile Shear	–
"	"	"	200	51,800	33,200		36.7	41.4	90% Cleavage & Ductile Shear	–
"	"	"	300	41,000	30,200		40.8	46.2	20% Cleavage & Ductile Shear	–
"	"	"	400	27,000	24,100		23.6	35.2	Ductile Shear	–
"	"	"	500	14,800	14,800		11.9	18.1	Intergranular	–
"	"	"	600	8,200	7,600		8.7	3.8	Intergranular	–
X84 Can C	2.3×10^{20}	280 – 435 300 – 500	–195	64,800			0.1	0.2	Cleavage	–
"	"	"	– 72	61,500			0.1	0.2	Cleavage	–
"	2.5×10^{20}	"	R.T.	65,700		Upper 65,700 Lower 63,500	0.5	0.2	Cleavage	–
"	"	"	200	55,500		Upper 55,500 Lower 51,200	22.8	34.1	92% Cleavage & Ductile Shear	3.60
"	"	"	300	40,600		39,800	33.0	41.1	60% Cleavage & Ductile Shear	3.06
"	"	"	400	31,600		Upper 31,600 Lower 29,000	18.3	29.2	Ductile Shear	0.33
"	2.3×10^{20}	"	500	19,900	19,700		9.3	18.0	Intergranular	–
"	"	"	600	9,700	9,600		7.3	2.9	Intergranular	–
"	Control	"	R.T.	88,300	43,300		15.3	16.6	95% Cleavage	–

"	"	"	200	51,000	33,600		34.0	38.5	80% Cleavage & Ductile Shear	–
"	"	"	300	36,800	25,100		27.6	40.4	20% Cleavage & Ductile Shear	–
"	"	"	400	25,700	21,300		20.3	35.1	Ductile Shear	–
"	"	"	500	18,000	17,500		13.7	14.9	Intergranular	–
"	"	"	600	6,600	6,200		5.8	2.3	Intergranular	–

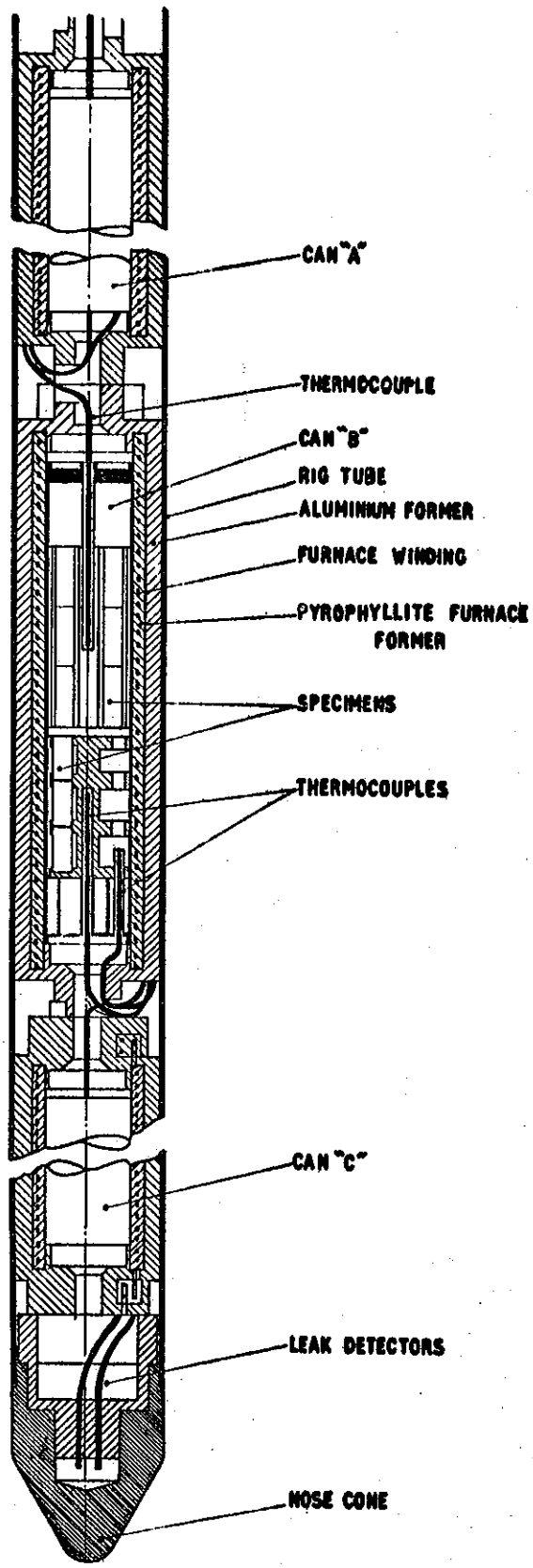

FIGURE 1 SCHEMATIC DIAGRAM OF HIGH TEMPERATURE IRRADIATION RIG

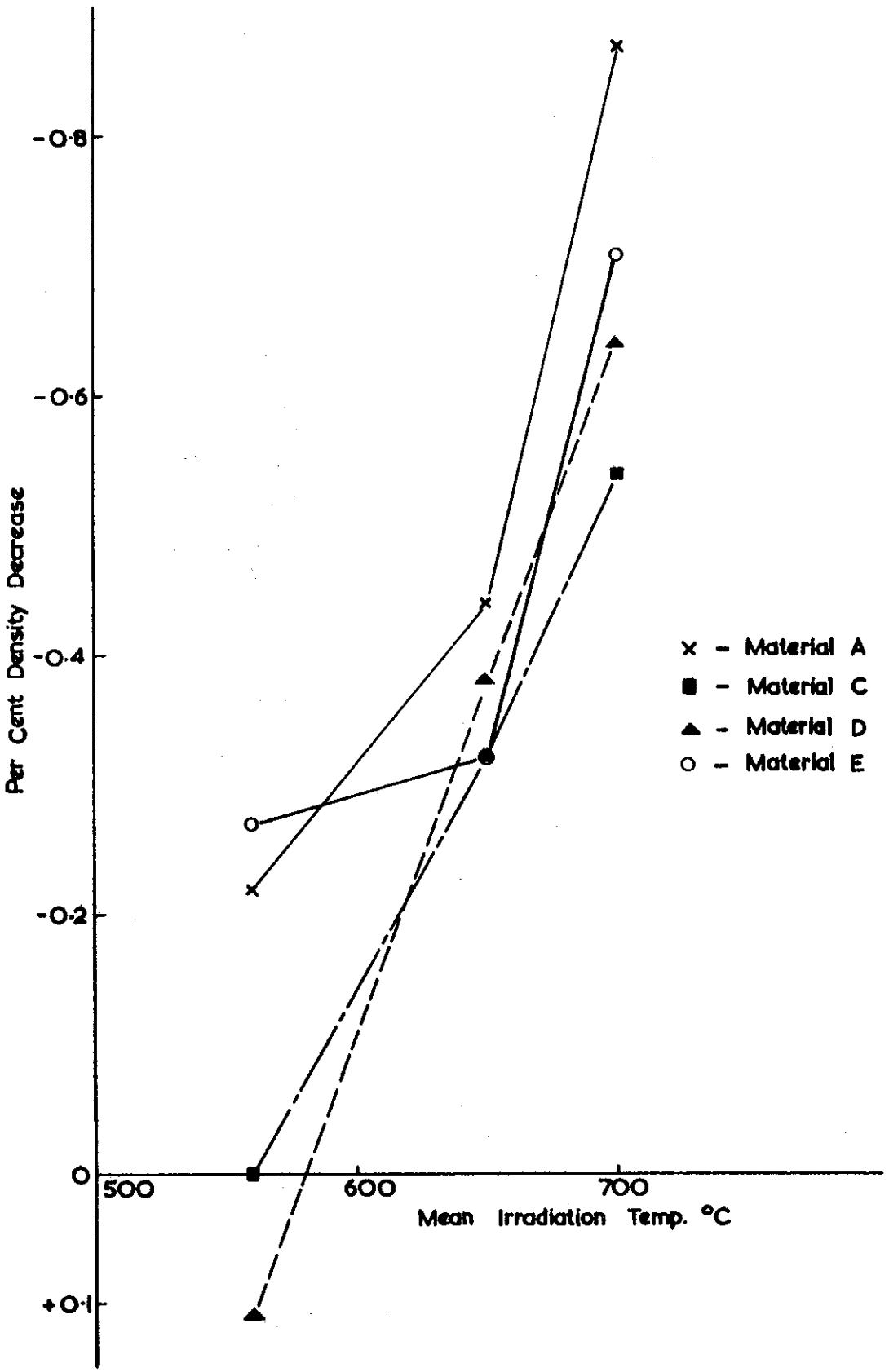

FIGURE 2 VARIATION OF DENSITY CHANGE WITH IRRADIATION TEMPERATURE FOR DOSES IN THE RANGE 4 TO 6 x 10^{20} nvt

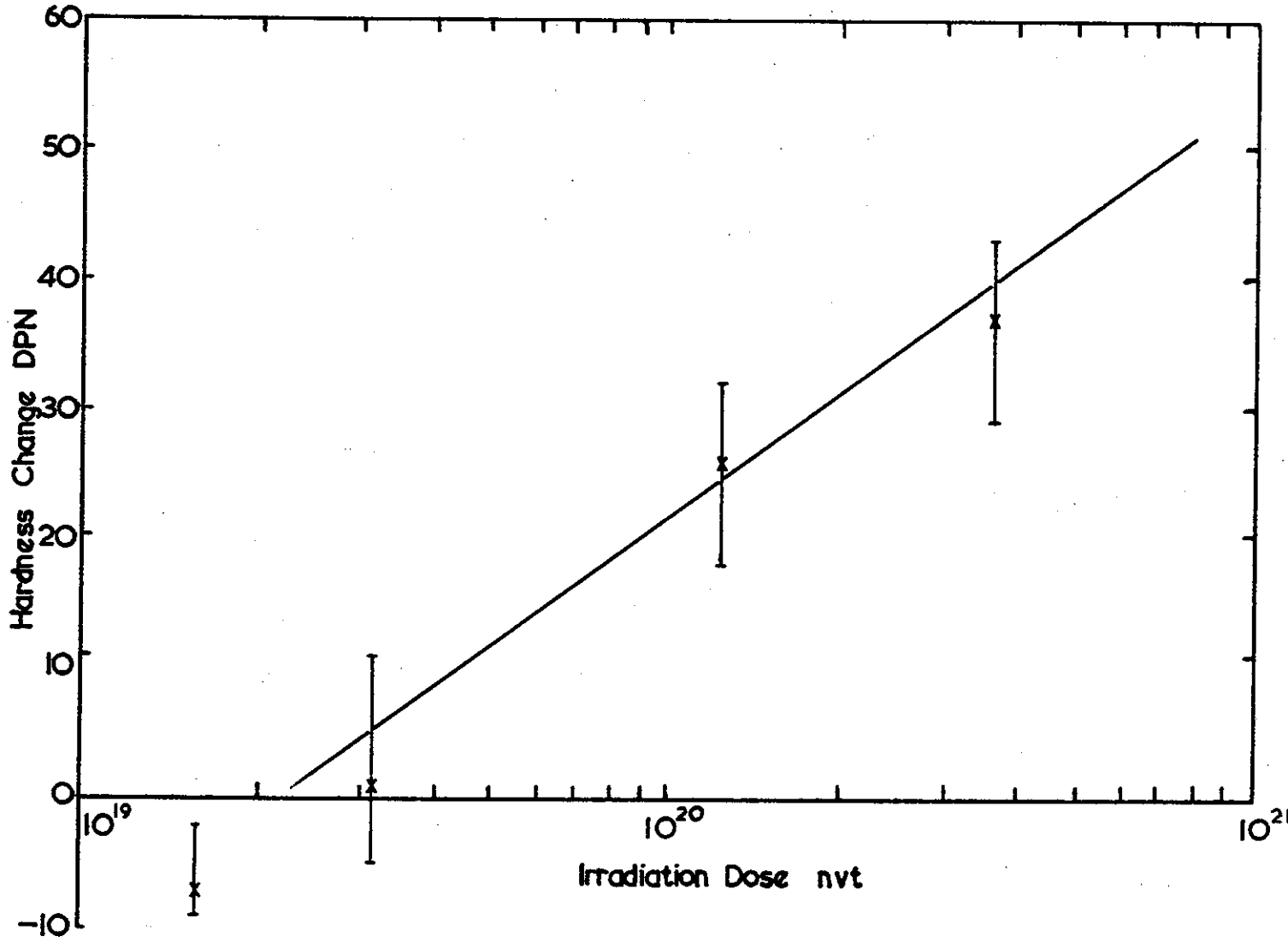

FIGURE 3 HARDNESS CHANGE VERSUS DOSE FOR IRRADIATION AT 75°C TO 100°C

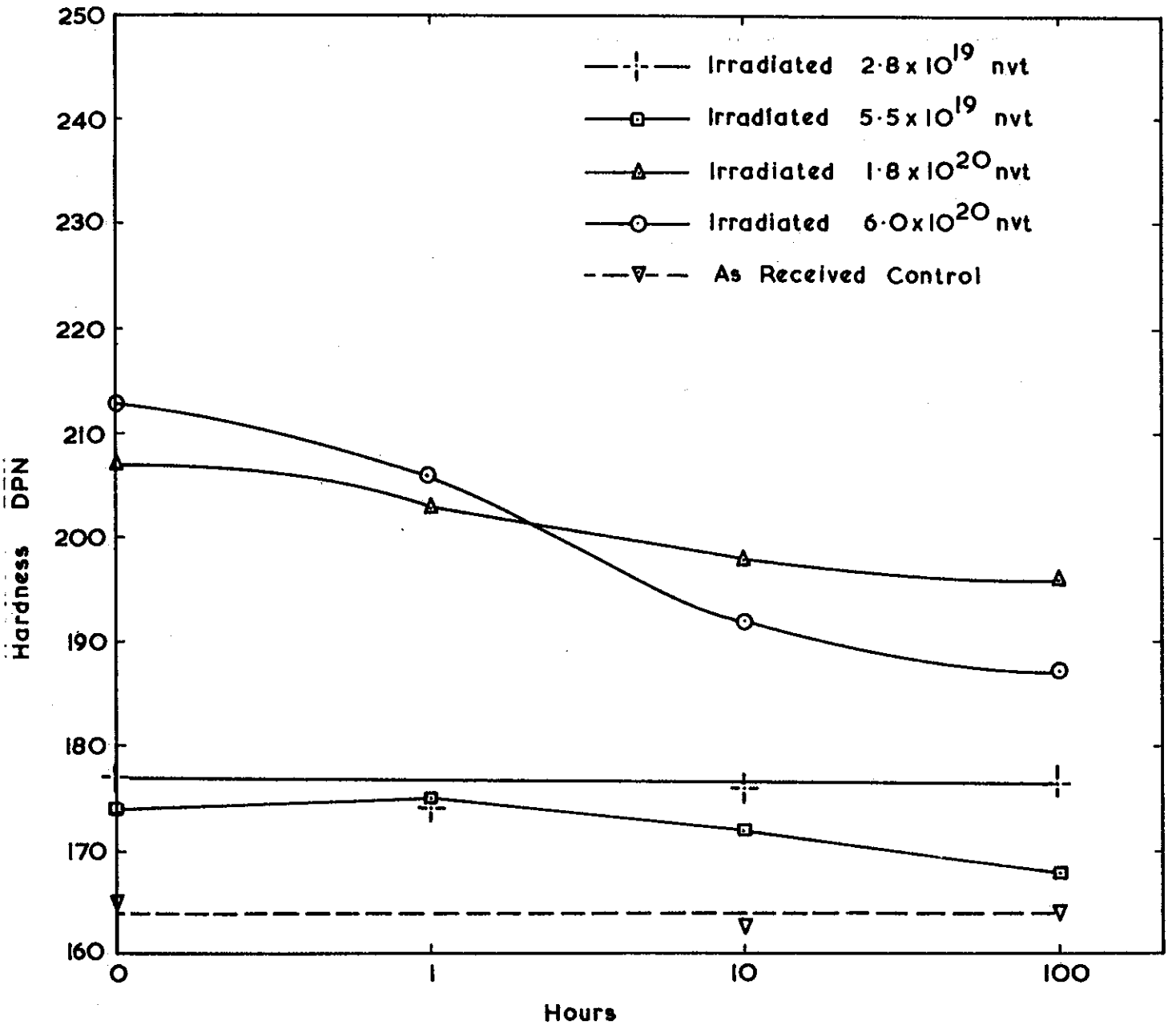

FIGURE 4

CHANGE IN HARDNESS WITH TIME FOR ANNEALING AT 600°C OF SPECIMENS OF MATERIAL A IRRADIATED TO VARIOUS DOSES

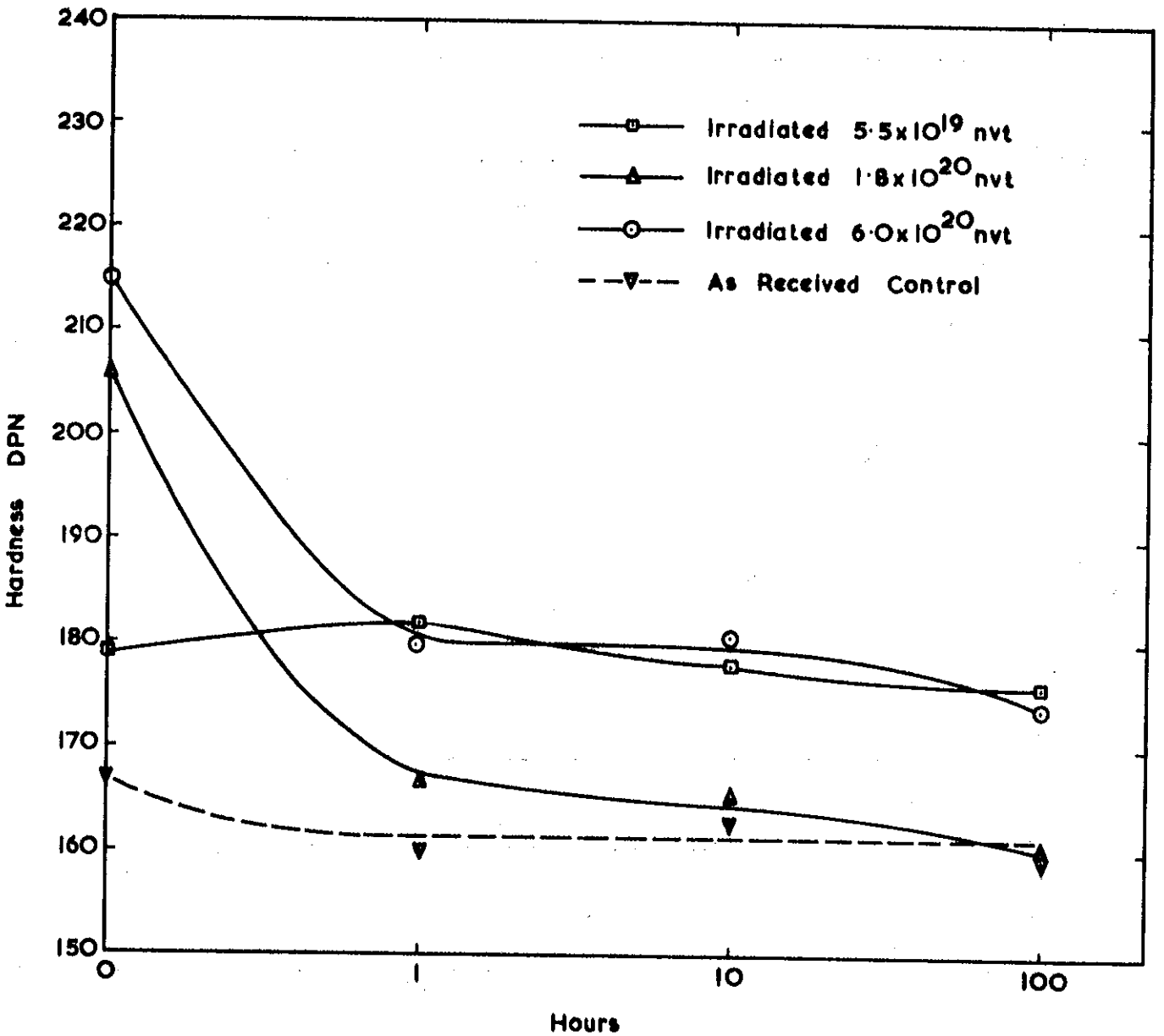

FIGURE 5
CHANGE IN HARDNESS WITH TIME FOR ANNEALING AT 800°C OF SPECIMENS OF MATERIAL A IRRADIATED TO VARIOUS DOSES

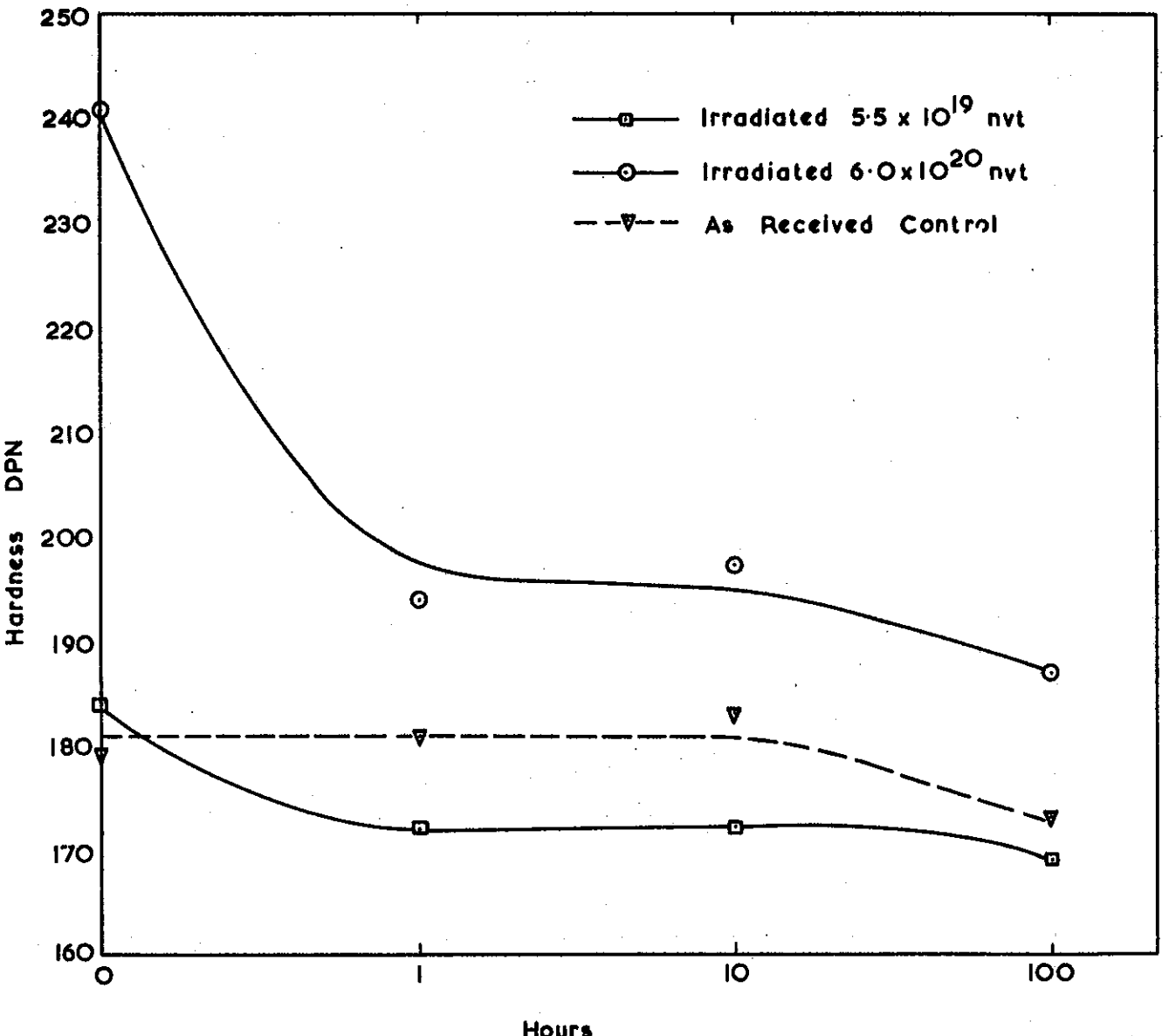

FIGURE 6
CHANGE IN HARDNESS WITH TIME FOR ANNEALING AT 1000°C OF SPECIMENS OF MATERIAL A IRRADIATED TO VARIOUS DOSES

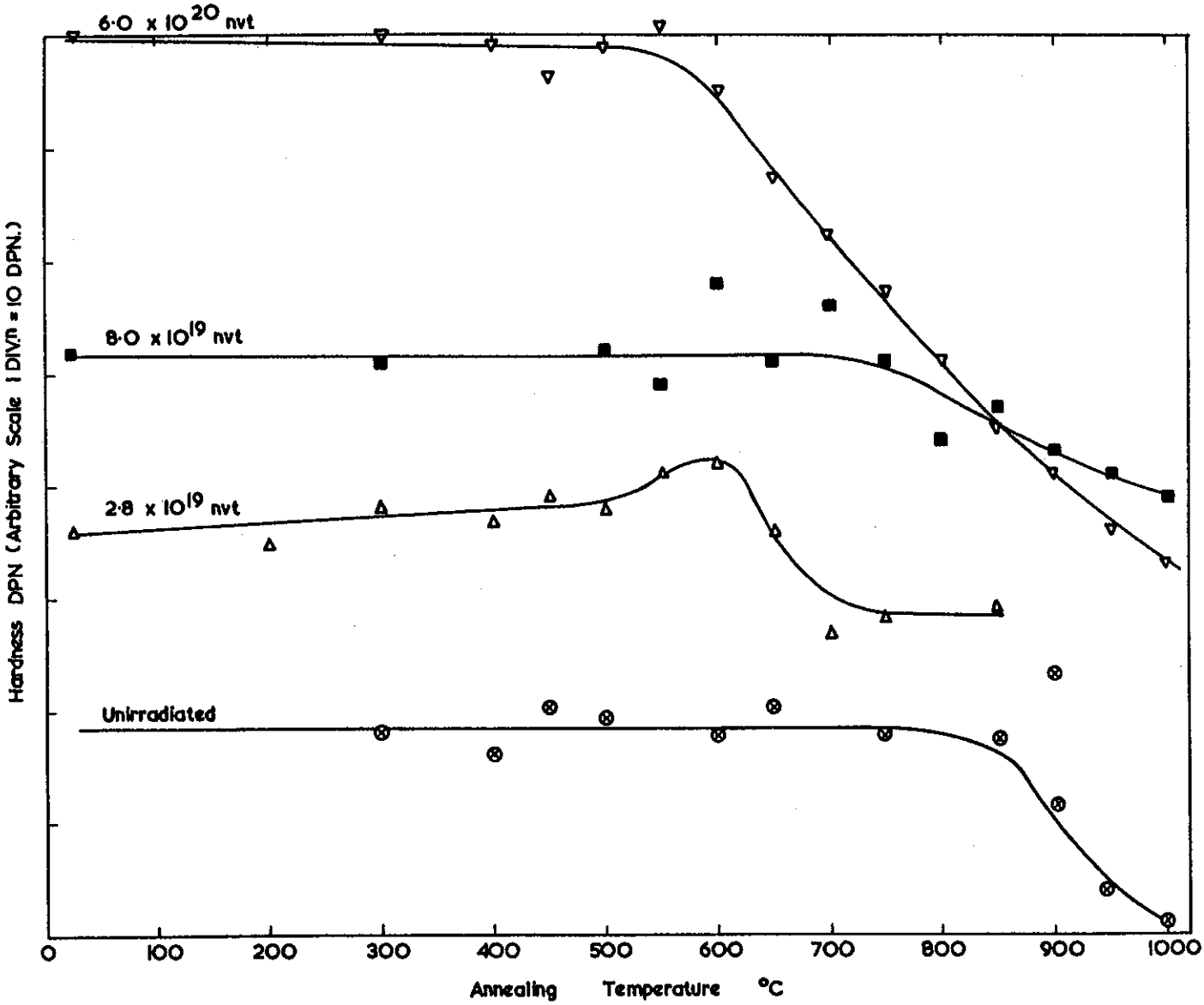

FIGURE 7
RECOVERY OF HARDNESS CHANGE VERSUS ANNEALING TEMPERATURE FOR ONE HOUR ANNEALS OF MATERIAL A IRRADIATED TO VARIOUS DOSES

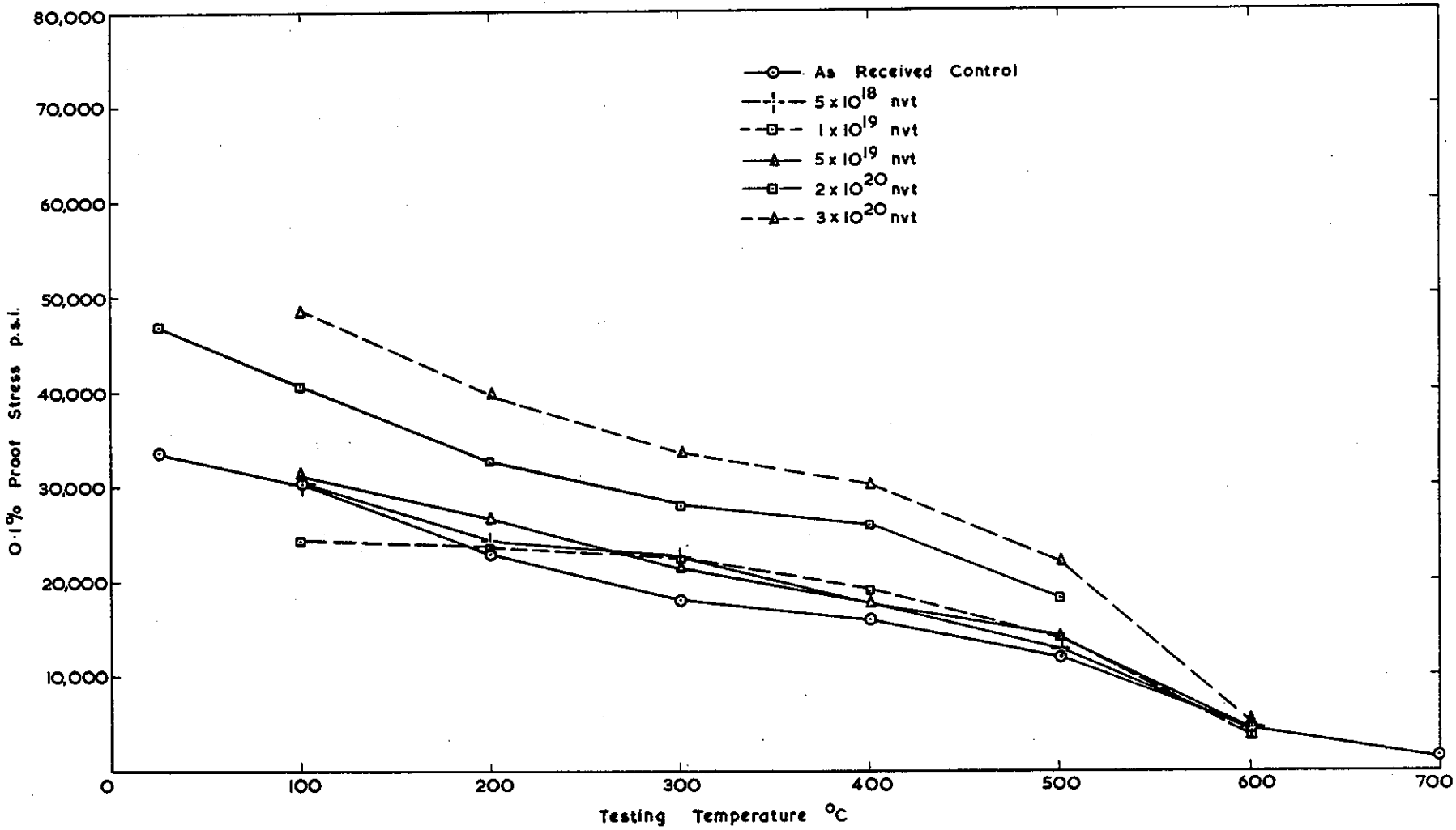

FIGURE 8 VARIATIONS IN 0·1 PER CENT. PROOF STRESS WITH TESTING TEMPERATURE IN MATERIAL A IRRADIATED TO VARIOUS DOSES AT 75°C TO 100°C

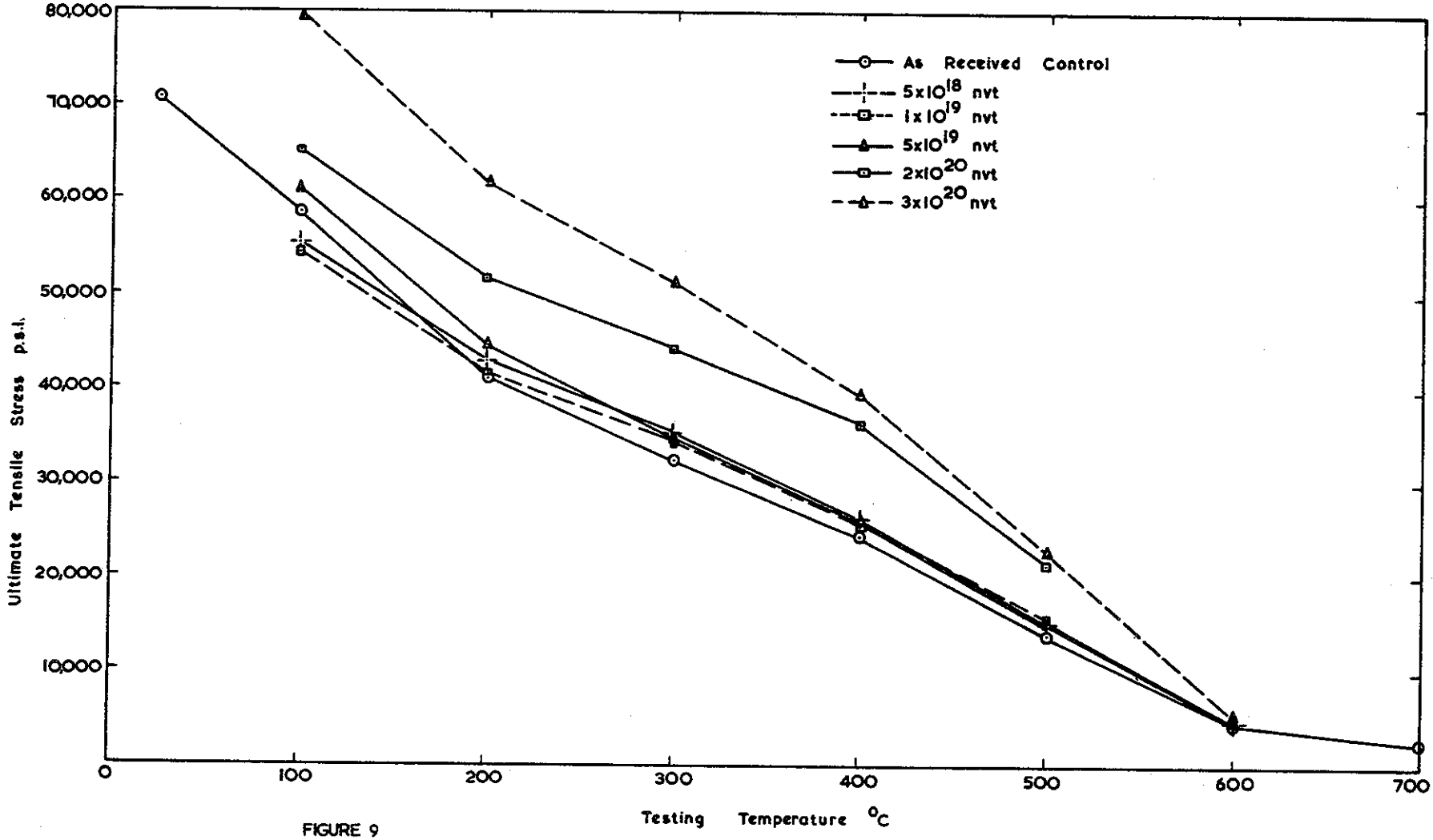

FIGURE 9

VARIATIONS IN ULTIMATE TENSILE STRENGTH WITH TESTING TEMPERATURE IN MATERIAL A IRRADIATED TO VARIOUS DOSES AT 75°C TO 100°C

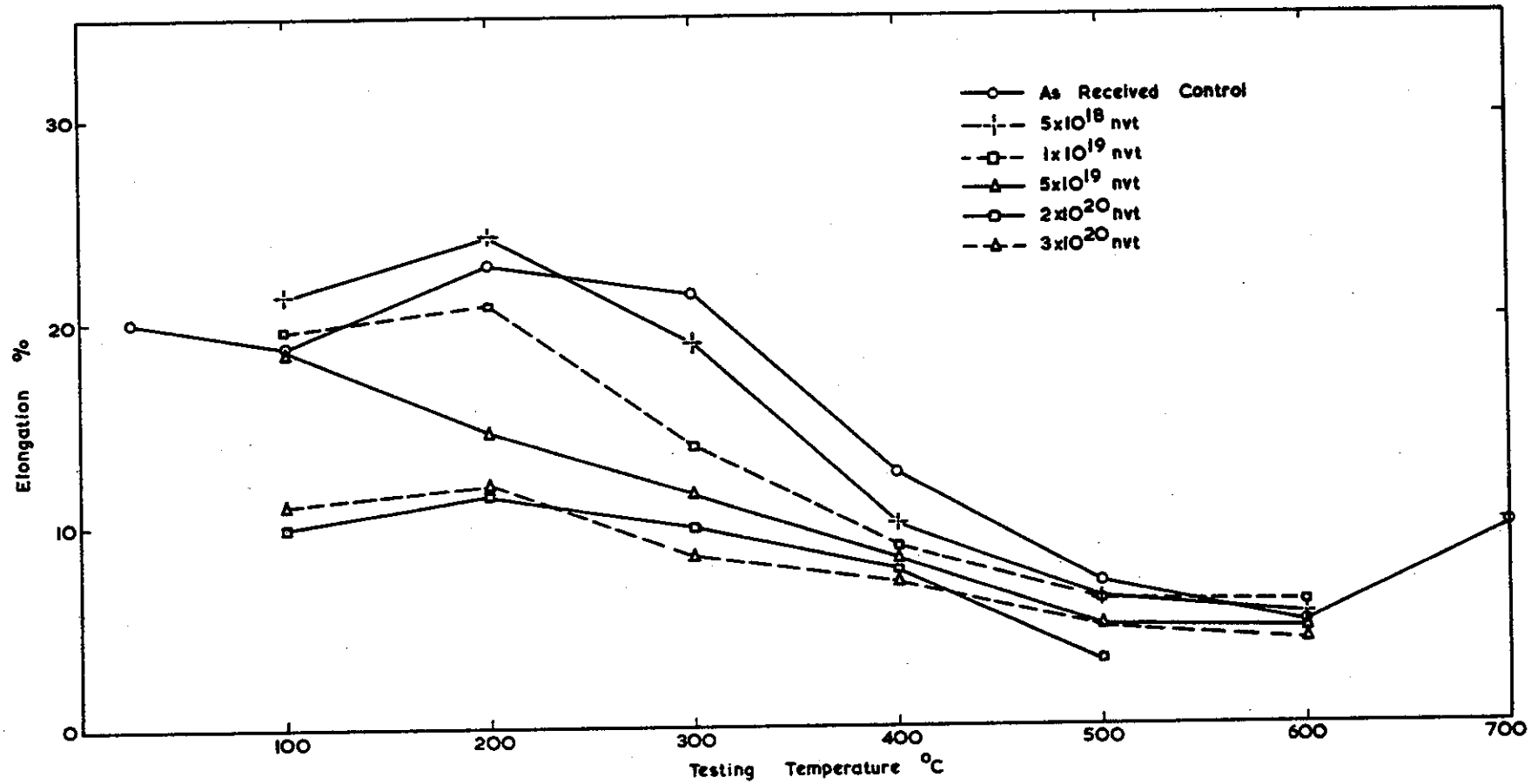

FIGURE 10 VARIATIONS IN ELONGATION WITH TESTING TEMPERATURE IN MATERIAL A IRRADIATED TO VARIOUS DOSES AT 75°C TO 100°C

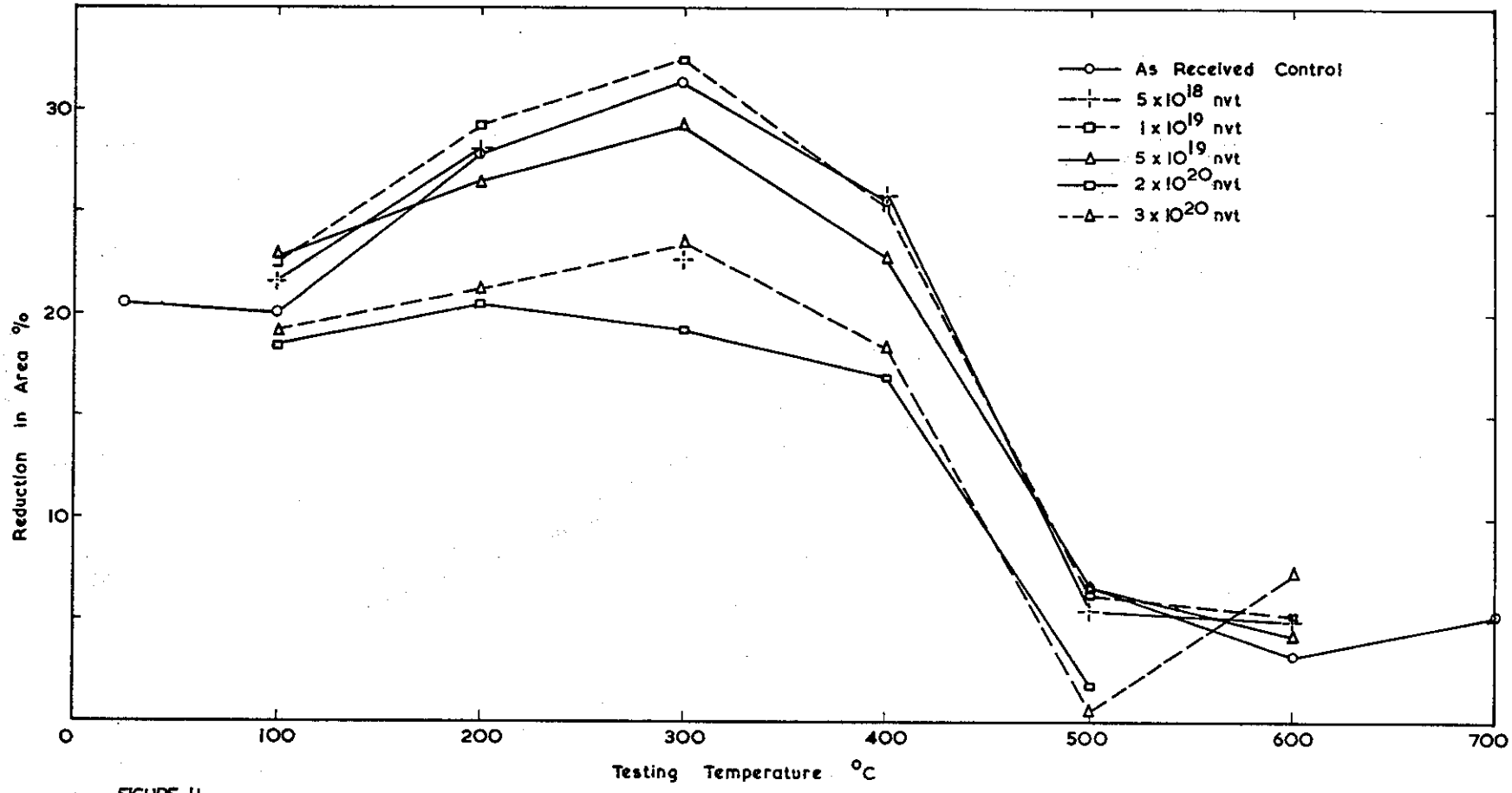

FIGURE 11 VARIATIONS IN REDUCTION IN AREA WITH TESTING TEMPERATURE IN MATERIAL A IRRADIATED TO VARIOUS DOSES AT 75°C TO 100°C

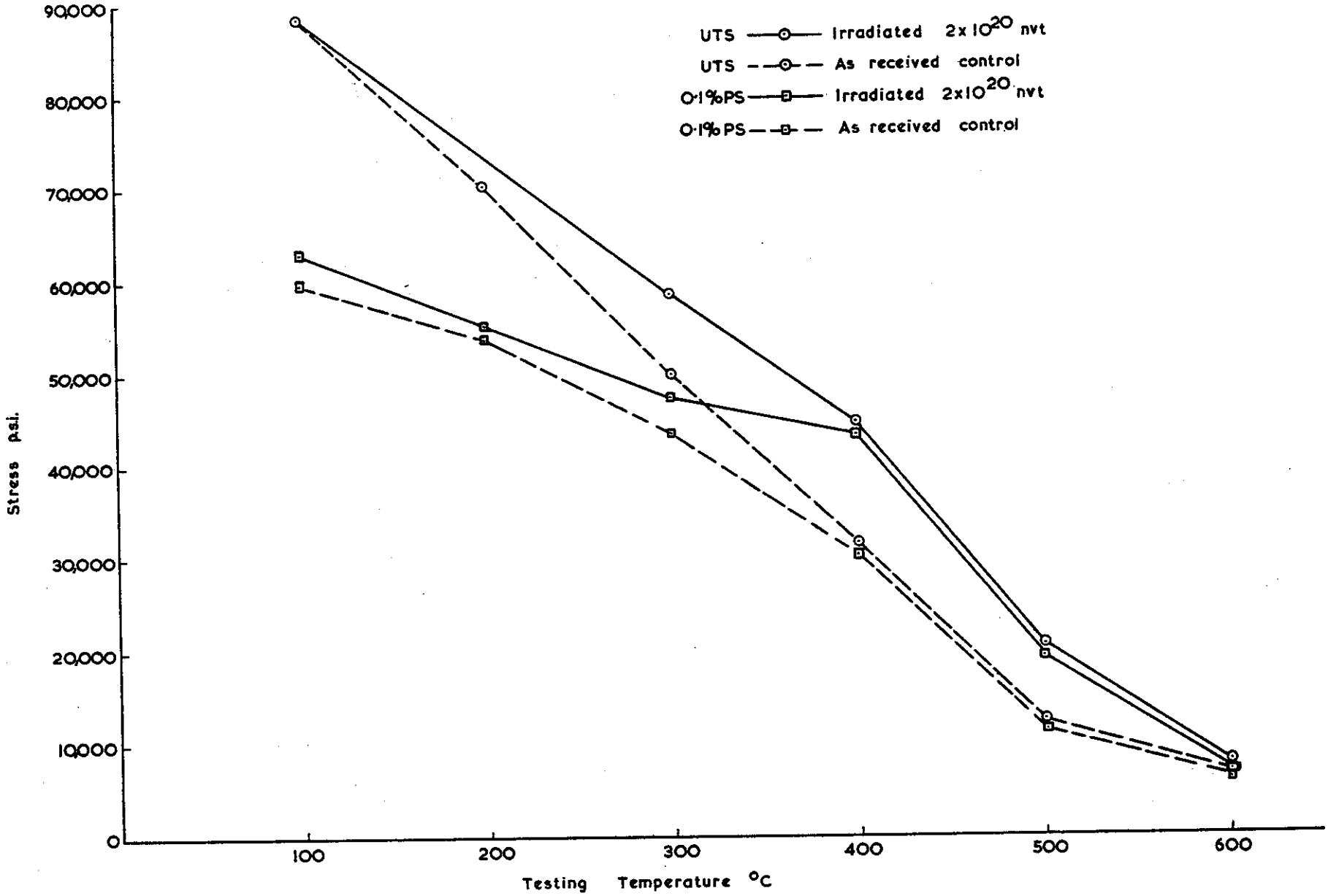

FIGURE 12
VARIATIONS IN 0·1 PER CENT. PROOF STRESS AND ULTIMATE TENSILE STRENGTH WITH TESTING TEMPERATURE IN MATERIAL B IRRADIATED TO 2×10^{20} nvt AT 75°C TO 100°C

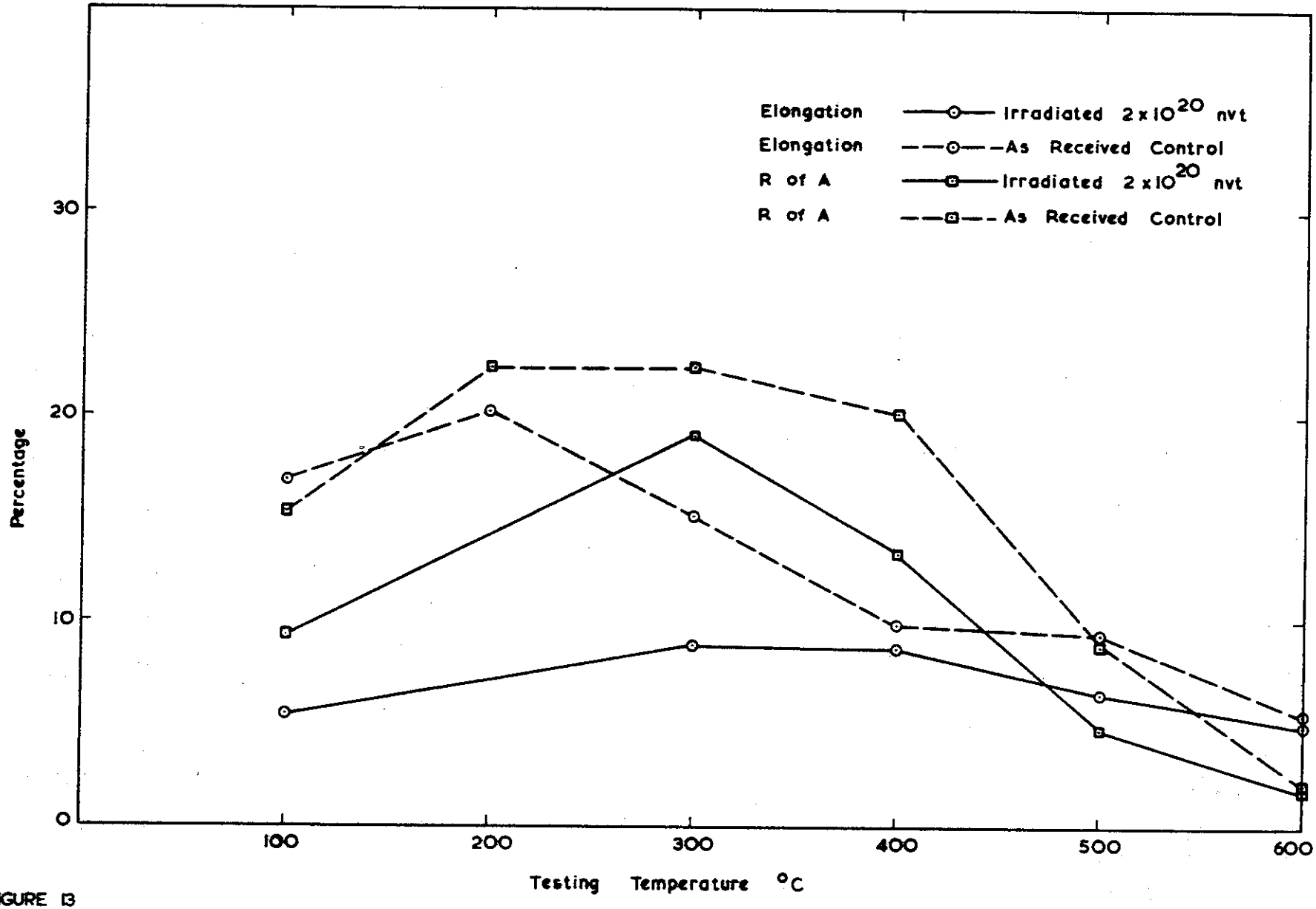

FIGURE 13

VARIATIONS IN ELONGATION AND REDUCTION IN AREA WITH TESTING TEMPERATURE IN MATERIAL B IRRADIATED TO 2×10^{20} nvt AT 75°C TO 100°C

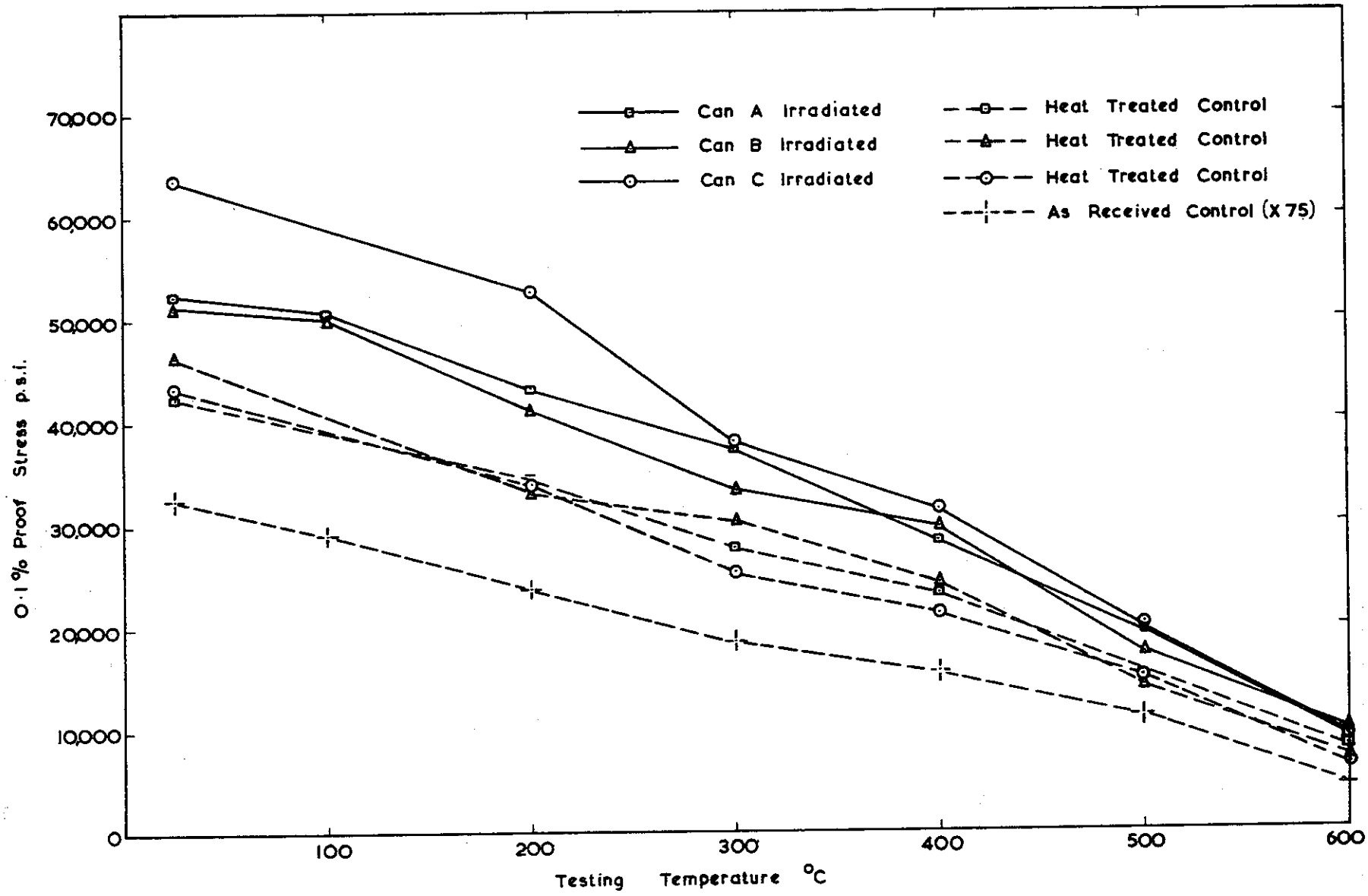

FIGURE 14

VARIATIONS IN 0·1 PER CENT PROOF STRESS WITH TESTING TEMPERATURE FOR MATERIAL A IRRADIATED AT ELEVATED TEMPERATURES

Irradiation conditions : Can A $1{\cdot}5\times10^{20}$ nvt at 500°C, Can B $2{\cdot}5\times10^{20}$ nvt at 600°C, Can C $2{\cdot}4\times10^{20}$ nvt at 300°C to 500°C

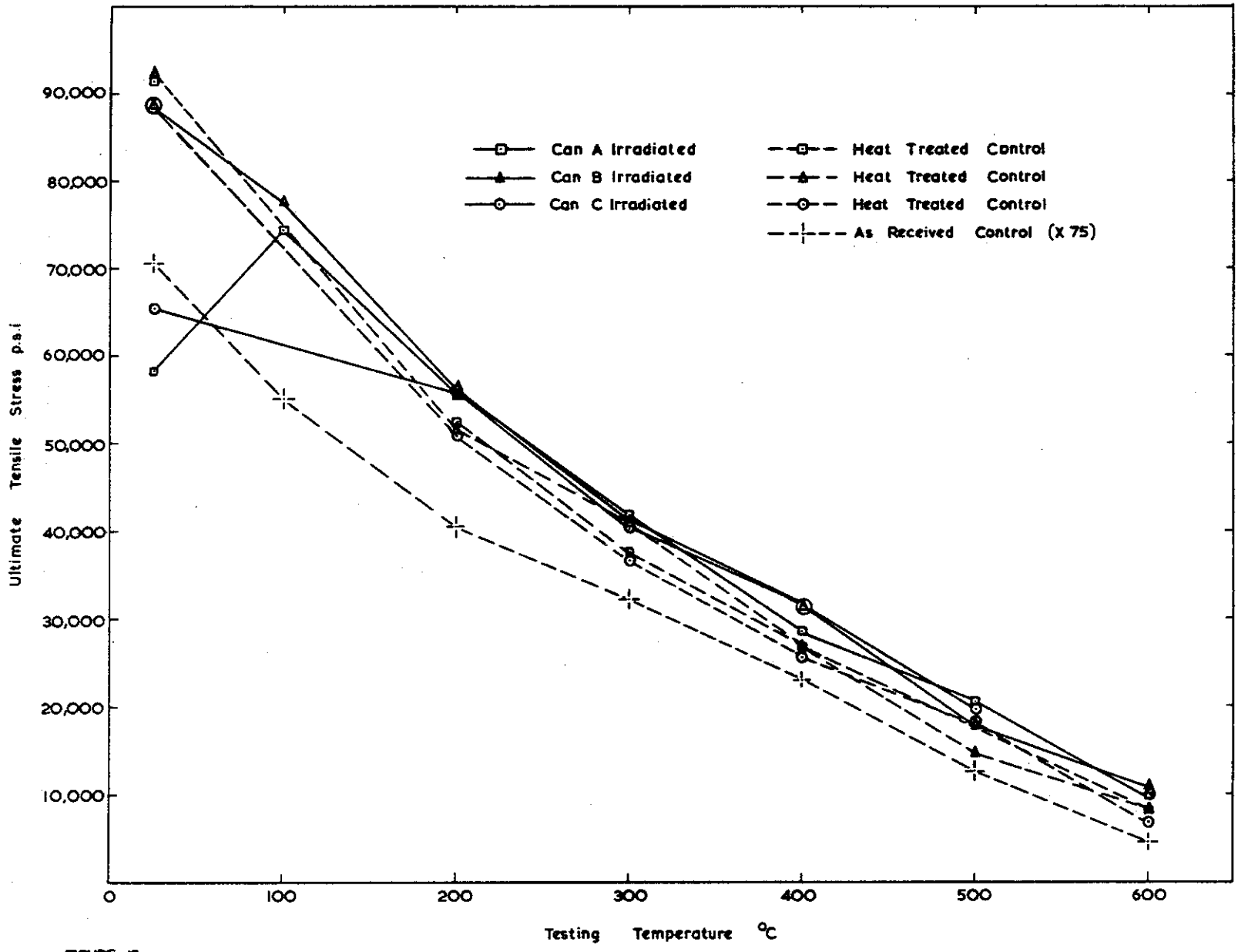

FIGURE 15

VARIATIONS IN ULTIMATE TENSILE STRENGTH WITH TESTING TEMPERATURE FOR MATERIAL A IRRADIATED AT ELEVATED TEMPERATURES

Irradiation conditions: Can A $1{\cdot}5 \times 10^{20}$ nvt at 500°C, Can B $2{\cdot}5 \times 10^{20}$ nvt at 600°C, Can C $2{\cdot}4 \times 10^{20}$ nvt at 300°C to 500°C

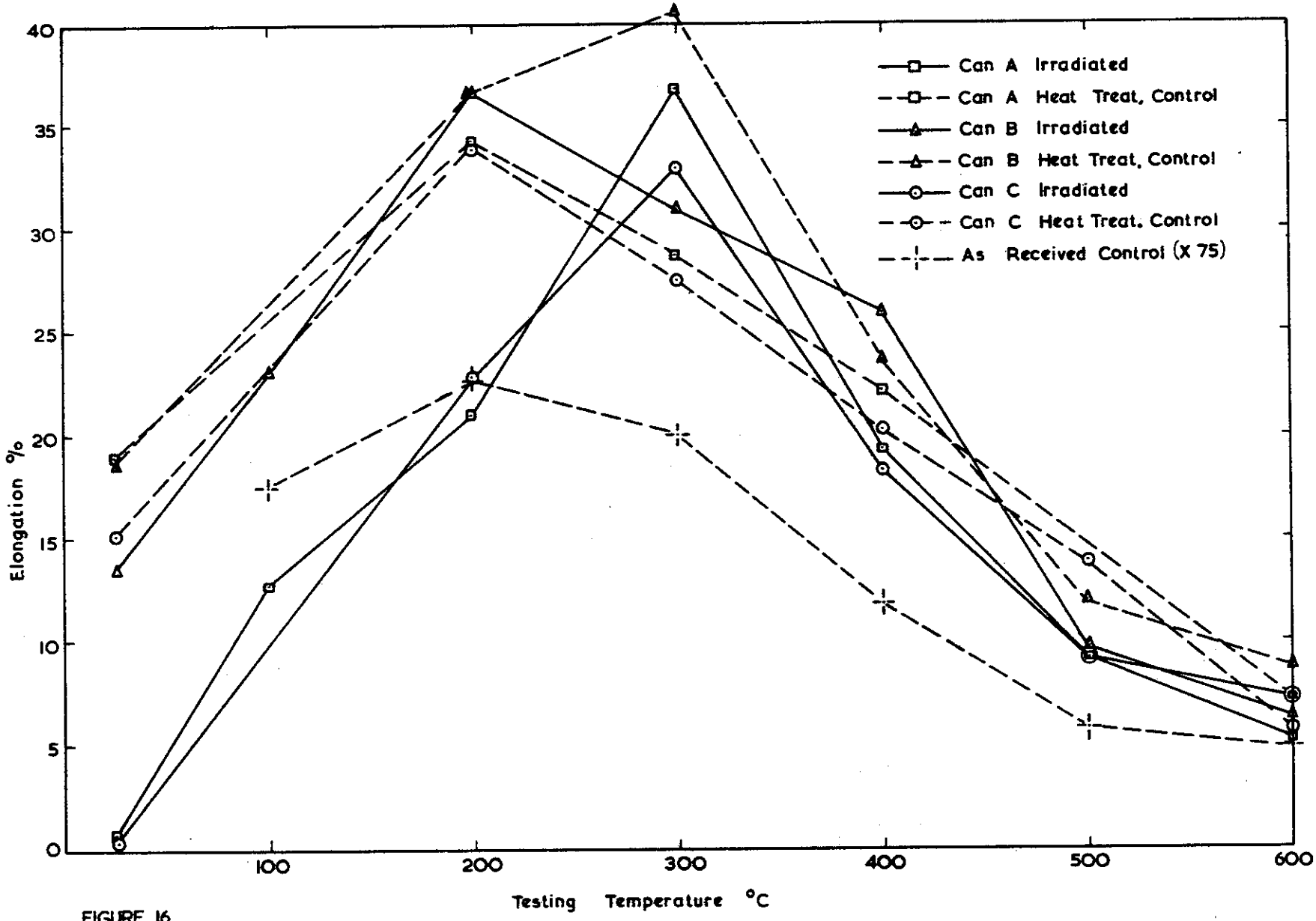

FIGURE 16

VARIATIONS IN ELONGATION WITH TESTING TEMPERATURE FOR MATERIAL A IRRADIATED AT ELEVATED TEMPERATURES

Irradiation conditions: Can A $1{\cdot}5 \times 10^{20}$ nvt at 500°C, Can B $2{\cdot}5 \times 10^{20}$ nvt at 600°C, Can C $2{\cdot}4 \times 10^{20}$ nvt at 300°C to 500°C

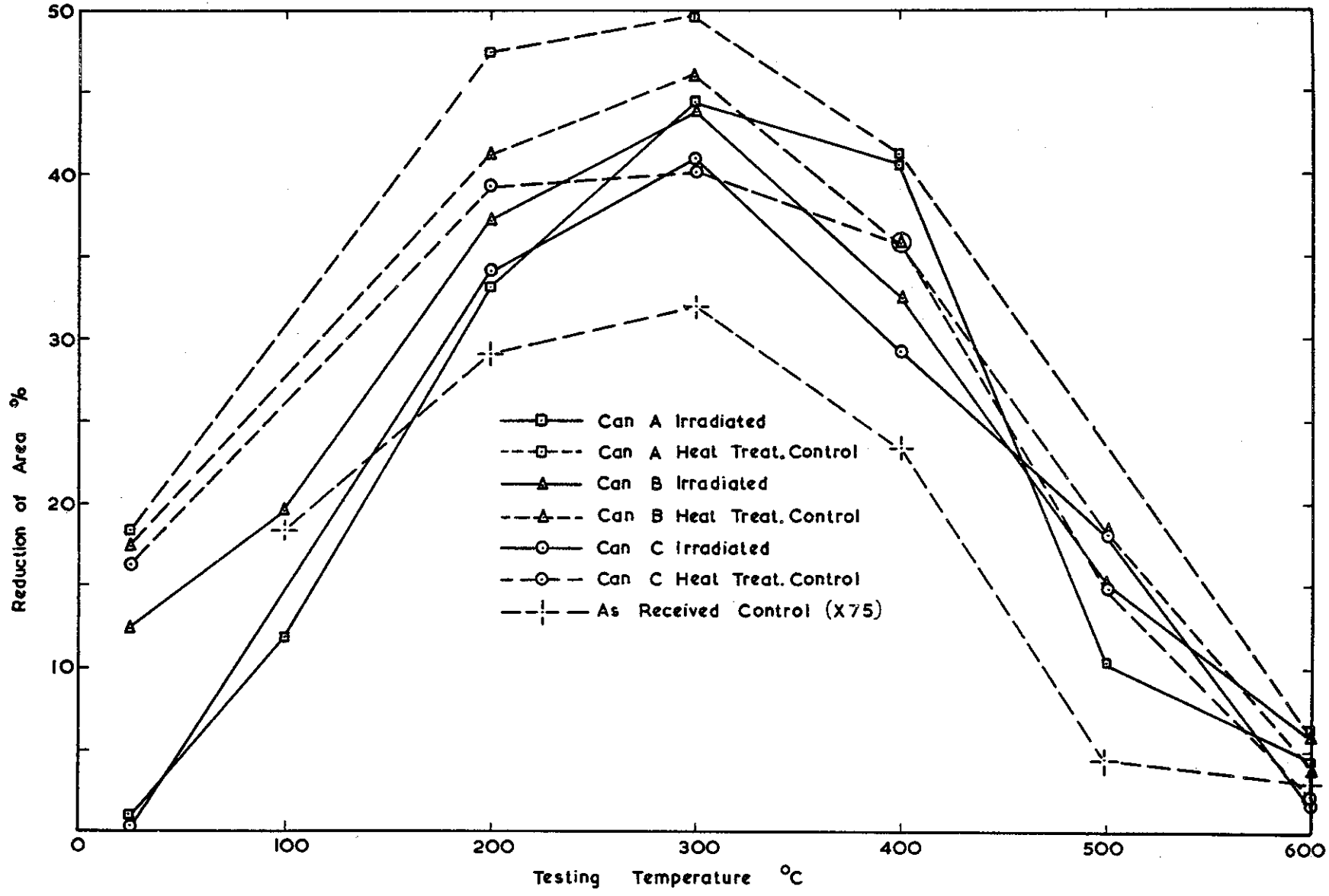

FIGURE 17

VARIATIONS IN REDUCTION IN AREA WITH TESTING TEMPERATURE FOR MATERIAL A IRRADIATED AT ELEVATED TEMPERATURES

Irradiation conditions : Can A $1{\cdot}5 \times 10^{20}$ nvt at 500°C, Can B $2{\cdot}5 \times 10^{20}$ nvt at 600°C, Can C $2{\cdot}4 \times 10^{20}$ nvt at 300°C to 500°C