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Berillium Irradiation Embrittlement Test (BSBE) Programme

Assessment of the Tensile and Fracture Toughness Test Data

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Abstract

An independent assessment has been made of the ambient and elevated temperature tensile and fracture toughness properties of unirradiated (reference), aged (~ 2000 h at temperatures in the range 185 to 605 C) and irradiated (2.1×10^{25} nm^{-2} (>1 MeV) at 200, 400 and 600 °C (nominal)) hot isostatic (HIP) and vacuum hot (VHP) pressed S-65 and S-200F beryllium grades as determined by SCK/CEN, Mol Belgium. The effects of material (powder consolidation method, beryllium oxide and elemental impurity contents and grain size), test (temperature) and irradiation (temperature, atom displacement dose and helium concentration) variables were analysed to further the development of composition - structure - property relationships for unirradiated and irradiated beryllium.

Although detailed microstructural examinations of the unirradiated and irradiated materials and other investigations are required to clarify some of the present uncertainties, the results of the analysis demonstrate that the tensile proof and ultimate strengths of the reference and aged beryllium grades at a given test temperature increase with the inverse square root of the grain diameter (d) in accordance with the Hall-Petch relationship. The tensile yield strengths are also determined by the impurity elements and precipitates which increase the friction stress opposing the movement of free dislocations in the lattice. The ductilities of the reference and aged beryllium peak at 230-310 °C whilst the non-uniform elongations generally decreases with increasing BeO (0.5 to 1.2%) and/or impurity contents at temperatures of 310 to 605 °C. These observations are consistent with a change in fracture mode from brittle, transgranular cleavage to ductile (dimple) fibrous and, possibly, intergranular with increasing test temperature.

The beryllium grades are severely embrittled at irradiation/test temperatures of ≤ 310 °C but some recovery of the tensile ductility occurs at ≥ 485 °C. There is an increased tendency to cleavage fracture at the lower temperatures, the cleavage stress of the irradiated beryllium being dependent on $d^{-½}$. The irradiated tensile samples fail by grain boundary and/or ductile dimple fracture at the higher temperatures, the intergranular fracture probably resulting from the stress - induced growth and coalescence of adjacent helium bubbles.

The fracture toughness values for the reference and aged beryllium are broadly in agreement with the tensile strengths at low and intermediate temperatures and increase with increasing reductions of a area at the higher temperatures. Irradiation markedly reduces the fracture toughness at all temperatures but there is some recovery at ~ 600 °C; the irradiated fracture toughness correlates reasonably well with the tensile proof stress and/or reduction of area values.

The beryllium grades do not differ significantly in their resistance to radiation damage at the lower test temperatures; however, the S-65 VHP and, in particular, the S-200F HIP grades are more ductile and marginally tougher at ≥ 435 °C

Versuchsprogramm Beryllium Versprödung unter Bestrahlung (BSBE): Auswertung der Zug- und Bruchzähfestigkeitstest

Zusammenfassung

Eine Auswertung der Zug- und Bruchzähfestigkeitstests von sowohl unbestrahlten als auch bestrahlten Beryllium-Proben wurde durchgeführt. Bei den analysierten Proben handelt es sich um hot isostatic pressed (HIP) S-65 und vacuum hot pressed (VHP) S-200F Beryllium, die bei 200 °C bzw. 400 °C und 600 °C bis zu einer Neutronenfluenz von $2.1 \bullet 10^{25}$ m^{-2} bestrahlt wurden.

Der Einfluß der Herstellungsparameter (z.B. Herstellungsmethode, Verunreinigungen, Korngröße usw.), sowie der Test- und Bestrahlungstemperatur auf den Materialeigenschaften wurde untersucht.

Obwohl weitere und detaillierte mikrostrukturelle Untersuchungen notwendig sind, um das Material vollständig zu charakterisieren, zeigen die Analysen, daß die Streckgrenze und die Bruchfestigkeit des unbestrahlten Materials bei einer konstanten Temperatur mit der Quadratwurzel der Korngröße zunehmen (Hall-Petch Gesetz).

Das Material wird durch die Neutronenbestrahlung bei Bestrahlungstemperaturen unter 310 °C stark versprödet. Eine kleine Wiedergewinnung der Streckbarkeit wurde jedoch bei Bestrahlungstemperaturen über 485 °C beobachtet.

Zusätzlich vermindert die Bestrahlung stark die Bruchzähfestigkeit des Materials bei allen Bestrahlungstemperaturen unter 600 °C und nur bei 600 °C wurde eine kleine Wiedergewinnung dieser Eigenschaft beobachtet.

Alle Beryllium-Proben verhalten sich mehr oder weniger gleich bei niedrigen Bestrahlungstemperaturen und sind alle spröde. Die S-65 VHP und die S-200F HIP Beryllium-Proben sind jedoch bei Bestrahlungstemperaturen über 435 °C etwas duktiler und zäher.

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D.R. Harries, M. Dalle Donne and F. Scaffidi - Argentina

Introduction

1. The specifications, manufacture and qualification of the powder metallurgy (PM) beryllium grades and the tensile, compact tension (C-T) and transmission electron microscope (TEM) disc samples for the BSBE Programme have been detailed [1]. The thermal ageing and irradiation procedures and conditions have been summarised [2][3] whilst the fast (> 0.1 and > 1 MeV) neutron fluences, displacements per atom (dpa) and helium concentrations, computed from the fission neutron fluences measured using dosimeters located adjacent to the beryllium specimens in the BR2 reactor irradiation rig, have been published [4]. The results of the ambient and elevated temperature tensile and fracture toughness tests on the unirradiated (reference), aged (thermal control) and irradiated specimens have been documented periodically [5 - 12] and the scanning electron microscope (SEM) observations of the fracture surfaces of the tensile samples presented [13]. However, there are errors in the tensile elongations quoted in some of the earlier publications but the ductility values are correctly reported in the more recent documents [10 - 12]; in addition, a final SCK / CEN, Mol report of the fracture toughness data is pending.

2. The results of an independent analysis and assessment of the tensile and fracture toughness data for the unirradiated (reference), thermally aged [2203 hours at six temperatures in the range 185 to 605 C] and irradiated [92.54 effective full - power days (2201 hours) in static NaK in BR2; nominal temperatures of 200, 400 and 600°C; 4.23 and 4.37 x 10^{25} n.m^{-2} (> 0.1 MeV) and 2.09 and 2.08 x 10^{25} n.m^{-2} (> 1 MeV) for the tensile and fracture toughness specimens respectively at the maximum flux plane of the reactor] beryllium grades in the BSBE Programme are described in this report. The principal characteristics of the materials investigated, produced by axial vacuum hot pressing (VHP) or direct hot isostatic pressing (HIP) of impact ground powders, are listed in Table I; the measured irradiation temperatures and computed atom displacement doses and helium concentrations for the tensile and fracture toughness specimens are given in Table II.

Table I

Oxide and Impurity Concentrations and Grain Sizes of the Beryllium Grades

Grade	BeO Content (wt. %)	Total Impurity (Fe, C, Al, Mg, Si, etc) Conc. (wt. %)	Average Grain Diameter (μm)
S-65. HIP	0.5	0.155	6.6
S-65. VHP	0.6	0.165	8.4
S-200F. HIP	0.9	0.29	7.1
S-200F. VHP	1.2	0.34	8.2

Table II

Temperatures, Displacement Doses and Helium Concentrations for the Tensile and Fracture Toughness Specimens Irradiated in the BR2 Reactor

Irradiation Temperature ^oC	Tensile Specimens dpa	Tensile Specimens appm He	Fracture Toughness Specimens dpa	Fracture Toughness Specimens appm He
185	1	300		
200			1.6	420
230			2.3	600
235	2.45	715		
310	0.8	240		
350			1.25	330
435			1.85	500
485	2.1	610		
540	2.5	750		
600	2.3	680	2.6	700
610			2.5	680

3. The primary aim of this exercise is to further the development of composition - structure - property relationships for unirradiated and irradiated beryllium. However, the realisation of this objective is inhibited to some extent because of (a) the relatively large number of material (powder consolidation process, BeO and elemental impurity contents and grain size), test (temperature) and irradiation (temperature, atom displacement dose and helium concentration) variables inherent in the BSBE irradiation embrittlement test programme, (b) the experimental and statistical uncertainties in the data as, in general, only one tensile specimen and one fracture toughness sample per condition were tested, and (c) the continued lack of detailed knowledge of the microstructural characteristics of the unirradiated and irradiated materials. These and other limitations of the experimental procedures, data and observations are evidenced and the additional investigations required to clarify some of the present ambiguities are outlined in this report.

Tensile Properties

4. The effects of ageing, irradiation and test (20 - 605^oC) temperature on the tensile 0.2% proof, ultimate (U.T.S.) and fracture (load divided by the cross - sectional area at fracture) stresses, uniform and total elongations and reductions of area of the unirradiated (reference), thermally aged and irradiated specimens of the S-65. HIP, S-65. VHP, S-200F. HIP and S-200F. VHP beryllium are detailed in Figs. A1, A2, A3 and A4 respectively in Appendix A. The tests were carried out at an initial strain rate of 2.5×10^{-4} s^{-1} except for the tests at 185 C on the unirradiated (reference) specimens which were fitted with extensometers; the initial strain rate in these tests was 5.5×10^{-4} s^{-1} up to 1% elongation and 1.25×10^{-3} s^{-1} thereafter. The influences of ageing and irradiation at 185 and 235 C on the ambient temperature properties and at 310, 485, 540 and 605^oC on the 250 C properties of the respective beryllium grades

are presented in Figs. B1, B2, B3 and B4 in Appendix B.

5. Tensile tests were also performed at ambient temperature and 400 C and initial strain rates of 2.5×10^{-4} and 8.33×10^{-5} s^{-1} on the round samples used in the BSBE Programme and the larger specimens employed by Brush - Wellman Inc. (BWI) (gauge lengths of 13.2 and 32 mm and diameters of 3.2 and 6.36 mm respectively) [11]. The tensile strengths and ductilities of the S-65C. VHP beryllium used in these tests are compared in Fig. 1 and show little or no effects of the initial strain rate and specimen dimensions within the ranges investigated.

Unirradiated (reference) and aged materials

6. The data plotted in the figures in Appendix A show that prior thermal ageing does not have a statistically significant influence on the proof and ultimate tensile strengths of the unirradiated (reference) beryllium. The strength values decline progressively with increasing test temperature, the magnitudes of the reductions decreasing in the order S-65 HIP (Fig. A1), S-200F HIP (Fig. A3), S-200F VHP (Fig. A4) and S-65 VHP (Fig. A2).

7. The uniform and total elongations and reductions of area of the unirradiated (reference) beryllium are all relatively small in the tests at ambient temperature, increase to maxima at about 300 C and then decrease at temperatures up to 605 C; the deformation at the higher test temperatures occurs predominantly in a non - uniform manner. The highest ductilities at temperatures ≥ 300 C are shown by the S-65. VHP grade (Fig. A2), followed by the S-65. HIP (Fig. A1) and S-200F. HIP (Fig. A3) materials. The ductilities (total elongations and reductions of area) of the reference S-200F. VHP beryllium (Fig. A4) are considerably lower than those of the other grades at all temperatures, being ≤ 3.5 and ~ 2% at 540 and 605 C respectively; the corresponding total elongation and reduction of area values for the other grades are ≥ 10 and ≥ 15%. Ageing does not significantly affect the uniform elongations of the reference beryllium but increases the total elongations and reductions of area of the S-200F. HIP (Fig. A3) and VHP (Fig. A4) beryllium at temperatures of 455, 540 and 605 C and of the S-65. VHP grade (Fig. A2) at intermediate temperatures (230 - 455°C). It follows that ageing enhances the non - uniform ductilities of these materials by modifying the ductile fracture process. The ductilities of the S-65. HIP beryllium (Fig. A1) are not influenced by the prior thermal ageing at all temperatures.

8. Thermal ageing at 185 and 230°C and at temperatures in the range 310 - 605°C has only a small or negligible effect on the tensile strengths and ductilities of the beryllium grades in tests at ambient temperature and 250°C respectively [Figs. B1, B2, B3 and B4 in Appendix B]. These data also confirm that the S-200F. VHP beryllium (Fig. B4) is significantly less ductile than the other grades at both ambient temperature and 250°C.

9. The present and other published data on the ambient and elevated temperature tensile properties of longitudinal specimens of S-65. VHP beryllium (analyses and grain sizes given in Table III) are compared in Fig. 2. There are relatively small differences in the proof stresses but the behaviour with respect to test temperature is similar (Fig. 2a); the U.T.S. data are, with few exceptions, in excellent agreement at all temperatures from ambient to 650 C (Fig. 2b). Whilst all the data show maximum ductilities at 300 - 400°C, the reduction of area values for the BSBE S - 65. VHP beryllium in the reference condition are considerably lower than those for the other lots at temperatures in the range 200 - 500°C. (Fig. 2c and d). However, prior thermal ageing enhances the ductilities of the BSBE beryllium such that the values

Table III

Analyses and Grain Sizes of S-65. VHP Beryllium

Grade	Lot No.	BeO wt. %	Impurity Content wt. % Fe	C	Al	Mg	Si	Ni	Grain Size μm	Ref.
S - 65C	4880	0.64	0.067	0.038	0.023	0.003	0.026	<0.002	9	14
S - 65C	4971	0.6	0.062	0.012	0.019	<0.01	0.016	0.013	9	15
S - 65B		0.98	0.09	<0.1	0.018	<0.003	0.026		18 - 20	16
S - 65C										17
S - 65C	4784	0.6	0.06	0.03	0.02	<0.01	0.03	<0.04	8.4	BSBE Prog.

approach or coincide with those of the other batches.

10. The 0.2% proof stresses and total elongations of the S - 200F. VHP beryllium tested in the BSBE Programme and in other investigations [17 - 19] are compared in Fig. 3a and b. The proof stresses are in reasonably good agreement at test temperatures up to about 500°C but the values for the BSBE lot are somewhat lower at higher temperatures. The total elongations for the BSBE material in the reference condition are also much lower at the higher test temperatures due to reduced non - uniform (necking) ductilities; however, thermal ageing again increases the high temperature elongations so that the values become comparable. The differences in behaviour are tentatively ascribed to differences in the BeO and impurity concentrations and distributions. The SEM examinations of the fracture surfaces revealed a high density of small BeO particles with which small pores are associated in the S - 200F. VHP BSBE lot [13]. In addition, the low ductility values at the high temperatures may be attributed to the high aluminium content (0.05 wt.%) as it is reported that this elemental impurity at the grain boundaries liquates and nucleates intergranular cracking at temperatures above 500°C [20]. This detrimental influence of the aluminium on the high temperature ductility may be negated by the formation of the higher melting point $AlFeBe_4$ intermetallic compound during the thermal ageing; it is often the practice to initially heat treat the beryllium at a temperature of about 870°C followed by step cooling to promote the formation of this phase [15]. However, analytical TEM examinations are required to clarify the reasons for the observed differences in behaviour and the effects of thermal ageing on the microstructures.

11. The absolute values and temperature dependences of the tensile 0.2% proof and ultimate strengths and ductilities (uniform and total elongations and reductions of area) for the unirradiated (reference) and thermally aged materials are also in good agreement, allowing for the disparities in the BeO and impurity contents (and, possibly, distributions) and the different test temperatures employed in the respective studies, with the reported data [20] for impact ground S - 65B. VHP (1.0% BeO), S - 200F. VHP (1.5% BeO) and S - 200F. HIP (1.5% BeO) beryllium.

12. The SEM examinations of the fracture surfaces show transgranular cleavage failure of the S-65. HIP specimens in the tests at ambient temperature [13]; ductile crack initiation precedes transgranular cleavage crack propagation at 230°C whilst ductile dimple fracture predominates

at the higher test temperatures. These observations are generally consistent with those reported in the literature [14][20]; failure at temperatures up to 200°C occurs by cleavage, ductile/ fibrous fracture takes place at 200 - 500°C, and some beryllium grades exhibit an increasing tendency to fracture intergranularly at higher temperatures.

13. The hexagonal close packed (hcp) beryllium crystal deforms by slip on the basal (0001) and prismatic ($10\bar{1}0$) planes (Fig. 4) at ambient and elevated temperatures. At temperatures below about 200°C the shear stress for the activation of prismatic slip is relatively high and slip on the basal planes is favoured (Fig. 5). Dislocations are piled up against a grain boundary as a result of coarse slip within the grain, resulting in an intense tensile stress field on one side of the slip band and compressive on the other side (Fig. 6). The tensile stress concentration may be alleviated by slip in the adjacent grain if it is suitably oriented, otherwise the combined tensile stress from the slip and the externally imposed stress can result in the initiation of a crack; the crack propagates rapidly to produce cleavage or pseudo - cleavage transgranular failure along the (0001) basal planes or is arrested following a reduction of the stress concentration at the crack tip by plastic flow. The critical stress for prismatic slip approaches that for basal slip with increasing temperature above 200°C and both slip systems become operative; the critical stress for cleavage fracture increases, the beryllium thus becomes more ductile and fails by fibrous fracture at temperatures up to 500 or 600°C.

14. The load - displacement (tensile stress - strain) curves are illustrated schematically in Fig. 7 and reflect the changes in the deformation and fracture modes of the beryllium with increasing test temperature (and after irradiation as discussed later). The stress - strain curves for the unirradiated (reference) and aged samples of all the beryllium grades tested at ambient temperature and 185 C are of type (b) [fracture after a small amount of plastic deformation] or (c) [fracture after significant uniform plastic deformation but with little or zero non - uniform (necking) elongation]. The stress - strain curves at the higher test temperatures (230 - 605°C) are generally of types (d), (e) or (f), with fracture occurring after significant non - uniform deformation following either extensive or limited uniform elongation. However, the unirradiated (reference) and thermally aged S-200F. HIP samples differ from those of the other grades in showing yield point behaviour as exemplified in the load - displacement curves at ambient temperature, 310 and 605 C reproduced in Figs. 8, 9 and 10 respectively. The main characteristics are as follows:

<u>Ambient Temperature</u>: The linear (elastic) region is followed by a yield point, with a relatively small yield point drop, and Lüders extension; there is significant uniform deformation and work hardening up to a maximum load at which fracture occurs with no non - uniform (necking) deformation.

<u>310°C</u>: The elastic portion is followed by distinct upper and lower yield points and a small Lüders extension; there is limited work hardening and little uniform deformation; the load decreases progressively to fracture with extensive non - uniform deformation (necking elongation) and reduction of area.

<u>605°C</u>: The linear elastic portion is followed by a very restricted work hardening region and limited uniform deformation; there is no indication of a yield point although serrated yielding is observed in some instances; the load decreases progressively to fracture with significant non - uniform ductility.

15. Both "normal purity" and high BeO content beryllium exhibit yield points [21]. The yield point behaviour has also been observed in tensile tests on S - 65C. VHP beryllium (0.6% BeO, 185 ppm Al, 620 ppm Fe, 130 ppm Ni, 160 ppm Si, 120 ppm C, others < 100 ppm each) at an initial strain rate of $1.1 \times 10^{-4} s^{-1}$ and temperatures in the ranges 100 - 415 C and 25 - 500 C for specimens machined in the longitudinal and transverse orientations respectively [15]; the detection of the upper and lower yield points was facilitated by the attachment of extensometers to the test samples in this investigation.

16. The yield point phenomenon is attributed to the locking of the dislocations by impurity atoms or precipitates of $AlFeBe_4$ or $FeBe_{11}$ as discussed in refs. [15][21]; in addition, the serrated yielding shown by the S-200F. HIP in the tests at 605°C (Fig. 10) may be due to dynamic strain ageing as a result of the transient locking of the glissile dislocations. However, the reason why the yield point phenomenon is not exhibited by some of the other beryllium grades tested in the BSBE Programme is not apparent at this stage.

17. It has been noted previously [21 - 23] that the tensile strengths of S-200F and other beryllium grades are dependent on $d^{-\frac{1}{2}}$, where d is the grain diameter. The tensile proof and ultimate stresses of the present beryllium grades at the respective test temperatures increase with decreasing grain size and can also be correlated with $d^{-\frac{1}{2}}$ as shown in Figs. 11 and 12. However, there is an indication that the 0.2% proof stress - $d^{-\frac{1}{2}}$ correlations differ for the S-65 and S-200F grades at the lower test temperatures, the data for the latter lying above those for the former. In addition, the U.T.S. - $d^{-\frac{1}{2}}$ relationship for the aged beryllium specimens appears to differ from that for the unirradiated (reference) samples at the higher test temperatures, particularly 540°C.

18. The Hall - Petch relationships between the tensile lower yield (σ_y) and brittle fracture (σ_f) stresses and d, originally derived for iron and mild steels, are as follows [24][25]:

$$\sigma_y = \sigma_i + k\, d^{-\frac{1}{2}} \quad \text{.......} \quad (1)$$

where σ_i is a measure of the friction stress opposing the movement of free dislocations in the lattice and can be divided into two terms [26]:

$$\sigma_i = \sigma_1 + \sigma_2 \quad \text{.......} \quad (2)$$

where σ_2 is the resistance of the lattice itself to dislocation movement,
σ_1 is the hardening caused by impurities, precipitates, other dislocations, clusters of point defects, helium bubbles, etc. in the lattice, and
k is a measure of the tensile stress required to initiate plastic yielding in the grains ahead of the plastic front and, hence, the dislocation locking stress which is normally sensitive to both the test temperature and rate of straining.

$$\sigma_f = \sigma_i + k_f\, d^{-\frac{1}{2}} \quad \text{.......} \quad (3)$$

where k_f is a measure of the tensile stress needed to crack the metal at the tip of a slip band or pile up of dislocations and is generally independent of temperature and rate of straining.

These situations have been depicted in Fig. 6.

19. The stress - strain curves for the S-200F. HIP beryllium samples exhibiting the yield point phenomenon have been analysed to determine the parameters σ_i and k by extrapolating the work - hardening portions of the stress - strain curves back to the elastic line as shown in Fig. 13. The work - hardening region is accurately described by the relation:

$$\sigma = K\varepsilon^n \quad \text{...} \quad (4)$$

where σ is the true stress, ε is the true strain and K and n are constants.

Thus, extrapolating the true stress - true strain work - hardening portion of the curves back to zero plastic strain enables the values of σ_i and k to be determined. The results show that the lower yield stresses are determined principally by σ_i which decreases progressively with increasing test temperature for both the unirradiated (reference) and thermally aged S-200F. HIP beryllium; the dislocation locking term is relatively independent of test temperature and prior history, the values of $kd^{-\frac{1}{2}} = \sim 20$ MPa being sensibly constant.

20. A linear correlation between the tensile total elongation at ambient temperature and the inverse square root of the grain diameter for beryllium has been reported [21] but such a correlation could not be established at any test temperature with the BSBE data.

21. Ductile fracture in tension occurs by the nucleation of voids as a result of the cracking or decohesion of hard particles in a soft matrix. The voids grow in three dimensions under the triaxial stress system established during the non - uniform (necking) deformation or at the root of a notch in tension. The voids eventually touch and the ligaments of matrix material extend so that each particle on the fracture surface lies in a dimple surrounded by edges and points. The rate of increase of void size is therefore dependent on the plastic strain, stress and stress state and the following expression has been derived for the critical limit to void growth for ductile fracture [27][28]:

$$\int 0.283 \exp.(3\sigma_m/2\sigma_{eq})\, d\varepsilon_p = \ln (R/R_0)_c \quad \text{....................................} \quad (5)$$

where σ_m and σ_{eq} are the mean and effective stresses, ε_p is the effective plastic strain and $(R/R_0)_c$ is the critical ratio of void growth for fracture.

22. The uniform and total elongations, the reduction of area values and the 0.2% proof stresses for the unirradiated (reference) beryllium at each test temperature are plotted as a function of the BeO content in Fig. 14. There is no systematic correlation of the ductility parameters with the BeO content at ambient temperature, 185 and 230°C; the beryllium specimens deform uniformly and fracture occurs in a relatively brittle manner without necking and the establishment of a triaxial stress system at these temperatures. Nevertheless, the non - uniform ductilities at the higher test temperatures (310 - 605°C) decrease with increasing BeO content in the range 0.6 - 1.2%; this effect is tentatively attributed to the progressively increasing influence of the BeO inclusions acting as cavity nucleation sites and thereby facilitating ductile fracture. The behaviour of the S-65. HIP grade, with a BeO content of 0.5%, does not appear to be entirely consistent with this hypothesis as the ductilities are generally lower than those of the S-65. VHP containing 0.6% BeO. However, the strengths of the S-65. HIP grade at the

relevant test temperatures are considerably greater than those of the S-65. VHP and the other beryllium grades, particularly at 540 and 605°C, thereby promoting ductile fracture and resulting in reduced ductilities. Furthermore, since the tensile strengths are proportional to $d^{-\frac{1}{2}}$, a fine grain size has an indirect effect in promoting ductile failure and lower ductilities at the higher test temperatures.

23. The effects of the BeO and impurity element contents on the reduction of area values for the unirradiated (reference) and thermally aged beryllium grades at test temperatures of 310, 455, 540 and 605°C are shown in Fig. 15. The enhancement of the non - uniform deformation and associated improvements in ductilities produced by the ageing are particularly evident in the grades with the higher BeO and impurity concentrations tested at 455, 540 and 605°C; these beneficial effects of ageing are tentatively attributed to the reduced segregation of the impurity atoms at the BeO - matrix and other interfaces as a result of their precipitation as $AlFeBe_4$ and/or $FeBe_{11}$ intermetallic compounds, thereby inhibiting micro - void growth and coalescence and increasing the resistance to ductile fracture.

24. Intergranular failures of metals and alloys at elevated temperatures generally occur by wedge or cavitation fracture. Wedge cracks are formed at grain boundary triple junctions as a result of the stress concentrations produced by grain boundary sliding if the applied shear stress (σ_S) exceeds a value given by the Stroh relationship [29]:

$$\sigma_S = [12\gamma G/\pi L]^{\frac{1}{2}} \quad \text{...(6)}$$

where γ is the effective surface energy for grain boundary fracture, G is the shear modulus and L is the length of the sliding boundary.

$$2\gamma = 2\gamma_S - \gamma_{GB} \quad \text{.. (7)}$$

where γ_S and γ_{GB} are the surface and grain boundary energies respectively

Assuming that L is determined by the grain diameter d, $\gamma = 1$ N.m^{-1} and G = 135 GPa, it is calculated that critical applied tensile stresses ($= 2\sigma_S$) of 560 and 495 MPa are required to nucleate intergranular wedge cracks in the present beryllium grades with grain diameters of 6.6 and 8.4 μm respectively. These stresses are well in excess of the ultimate tensile strengths and fracture stresses at test temperatures of $\geq$ 500 C and consequently grain boundary wedge cracking would not be expected in the present beryllium grades.

25. There are several possible nucleation sites for grain boundary cavitation failures during testing at elevated temperatures, the favoured being non - wetting particles. The cavities grow by grain boundary vacancy diffusion and eventually coalesce to form intergranular cracks if the applied tensile stress (σ_t) exceeds a value given by [30]:

$$\sigma_t = 2\gamma/r \cos^2\beta \quad \text{.. (8)}$$

where r is the radius of the cavity and β is the angle between the normal to the grain boundary and the axis of the tensile stress.

This reduces to $\sigma_t = 2\gamma/r$ for grain boundaries normal to the applied tensile stress direction.

Again taking $\gamma = 1\ N.m^{-1}$, it follows that cavities with radii of ≤ 20 nm lying on the transverse boundaries will grow at temperatures at which grain boundary vacancy diffusion is sufficiently rapid if the applied tensile stresses are ≥ 100 MPa. Thus, small non - wetting BeO particles and pores, high densities of which have been detected in the S-200F. VHP beryllium [13], could act as cavity nuclei for grain boundary fracture during the high temperature tensile testing. However, other intergranular precipitates can inhibit the grain boundaries acting as perfect sources and sinks for vacancies [31], in which case plastic growth by dislocation creep may become the dominant cavity growth process [32].

Irradiation effects

26. The effects of irradiation and test temperature on the tensile strengths and ductilities of the respective beryllium grades are included in Figs. A1 to A4 and B1 to B4 in Appendices A and B respectively.

27. The data in Figs. A1 to A4 show that all the beryllium grades exhibit significant radiation hardening (as evidenced by the increases in proof and ultimate tensile stresses) at irradiation / test temperatures of 185 to 485 or 540°C and severe embrittlement (decreases in the uniform and total elongations and reductions of area) over the whole irradiation / test temperature range investigated. The hardening is most pronounced at the lower temperatures of 230 and 310°C; progressive recovery of the radiation damage occurs with increasing irradiation / test temperature so that the proof and ultimate stresses are either unaffected (Figs. A2, A3 and A4) or reduced at 540 and about 600 C in the case of the S-65. HIP beryllium (Fig. A1). Partial recovery of the ductilities of the S-65. HIP (Fig. A1), S-65. VHP (Fig. A2) and S-200F. HIP (Fig. A3) beryllium also occurs at 485 and/or 540°C. but the total elongations of the S-200F. VHP grade remain at $\leq 1\%$ at these temperatures. The elongations and reduction of area values of all grades, apart from the S-200F. VHP beryllium which exhibits low ductilities in the unirradiated (reference) condition, are again reduced on testing at the higher temperatures of 540 and/or 600°C. The maximum radiation hardening and embrittlement are generally produced at about 310°C. even though the displacement doses and helium concentrations induced during the irradiation at this temperature are minimal (Table II).

28. All the tensile stress - strain curves for irradiation / test temperatures of 230 and 310°C are of type (a) (Fig. 7) [fracture within the elastic range] whilst those for the beryllium grades irradiated and tested at 185 C are either of type (a) (S-65. HIP), type (b) (S-65. VHP) or (c) (S-200F. HIP and VHP) [fracture after a small amount of uniform plastic deformation but without any necking]. The specimens irradiated and tested at the higher temperatures of 485, 540 and 600°C show plastic instability; the stress - strain curves are mainly of types (e) or (f) [fracture after limited uniform elongation but significant non - uniform deformation] although some samples exhibit type (c) (S-200F. VHP and S-65. HIP at 485 and 540°C respectively) or (d) (S-65. VHP at 540°C) [fracture after appreciable uniform and non - uniform deformation].

29. The specimens irradiated and tested at temperatures up to 485°C fail primarily by brittle cleavage fracture but with some intergranular decohesion in the S-200F. VHP beryllium [13]; pronounced intergranular cracking and isolated regions of ductile dimple (micro void coalescence) are evident in the fracture surfaces of the samples following reactor exposure and testing at higher temperatures.

30. There is little to choose between the various grades with respect to radiation damage

resistance at the lower temperatures ($\leq$ 310 C) as they are all very brittle. However, the ductilities of the S-65. HIP beryllium are marginally superior at 485 C whilst the S-65. VHP and S-200F. HIP grades are slightly more ductile at 540 and ~ 600 C.

31. The data in the figures in Appendix B show that irradiation at 235°C produces large increases in the proof and ultimate tensile strengths of the S-65. VHP beryllium in the room temperature tests whilst the reactor exposures at 185 and 235°C also reduce the ductilities to virtually zero. The irradiations at temperatures in the range 310 to 600°C also increase the tensile strengths and decrease the ductilities in the tests at 250°C but the magnitudes of the hardening and embrittlement are reduced following irradiation at 600 C. The influence of the irradiations at 185 and 235°C on the room temperature strength properties are not as pronounced for the S-65. HIP beryllium but the ductilities are again reduced to very low values. The post - irradiation ductilities of the S-65. HIP material at 250°C are marginally superior to those of the S-65. VHP grade except after exposure at 600°C whilst the magnitudes of the irradiation embrittlement are smaller due to the lower unirradiated non - uniform ductilities of the former. The ambient temperature ductilities of the S-200F. HIP and VHP grades are also severely reduced after irradiation at 185 and 235°C, whilst significant hardening and loss of ductility at 250°C occur following irradiation at the higher temperatures; the magnitudes of the hardening decrease with increasing irradiation temperature and there is again some recovery of the ductility of the HIP grade after exposure at 600°C.

32. Most of the previously published data on the effects of irradiation on the mechanical properties of beryllium was generated on grades which are now obsolete; these earlier radiation hardening and embrittlement data have been reviewed elsewhere [33]. However, the effects of neutron irradiation on current beryllium products, including Brush Wellman VHP S-65C (0.64% BeO) [14], S-65B (0.98% BeO) [16] and S-200F (1.2% BeO) [34] as well as various Russian grades ($\leq$ 1.2% BeO) [35][36], have also been investigated in recent years. The materials were irradiated in mixed spectrum fission reactors to displacement doses of $\leq$ 3 dpa and helium concentrations of $\leq$ 1140 appm at temperatures in the range 110 to 650°C [14][16][34] and in the BOR-60 fast reactor to 5 - 10 dpa and an average helium concentration of about 500 appm at temperatures of 350 to 800 C [35][36]. The trends in the data obtained in the post - irradiation tensile and bend tests at temperatures ranging from ambient to 800 C are generally similar to those observed in the BSBE Programme.

33. The radiation hardening and embrittlement at the lower irradiation / test temperatures result from the formation of vacancy and interstitial point defects, defect clusters, dislocation loops and intragranular helium bubbles. Theoretical considerations based on the cutting of the irradiation - induced obstacles by dislocations have shown that the lattice hardening is initially proportional to the square root of the neutron fluence (ϕt) or displacement dose (dpa), this dependency being obtained essentially as follows [37][38]:

If N is the number of clusters per unit area on the slip plane and l is the average spacing of the clusters, then:

$$l = 1 / N^{\frac{1}{2}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (9)$$

The stress (σ_c) required to move a dislocation is given by:

$$\sigma_c \propto Gb / l \quad \ldots\ldots (10)$$

where b is the Burgers vector of the dislocation.

Thus:

$$\sigma_c \propto GbN^{\frac{1}{2}} \quad \ldots\ldots (11)$$

N is proportional to the neutron fluence (or displacement dose) in the early stages of the irradiation so that the increase in stress ($\Delta\sigma_c$) required to move the dislocation is:

$$\Delta\sigma_c = A(\phi t)^{\frac{1}{2}} \text{ or } B(\text{dpa})^{\frac{1}{2}} \quad \ldots\ldots (12)$$

where A and B are constants.

However, the number of new obstacles formed during a given increment of fluence (or displacement dose) decreases as the fluence increases so that a saturation effect is obtained.

34. It has also been proposed [39] that the increased yield stress due to the pinning of the glissile dislocations by the intragranular helium bubbles is given by the following relationship:

$$\sigma_{ib} = \sigma_u + Gb\,(N_b r_b)^{\frac{1}{2}} \quad \ldots\ldots (13)$$

where σ_{ib} is the yield strength of the irradiated beryllium, σ_u is the unirradiated yield strength, N_b is the number of bubbles per unit volume and r_b is the radius of the intragranular bubbles.

This relationship has been successfully applied to predict the effects of irradiation on the yield strengths in earlier studies on hot pressed and extruded beryllium [40].

35. The 0.2% proof stresses for the unirradiated beryllium and for the irradiated beryllium grades exhibiting measurable plastic deformation prior to failure, together with the fracture stresses for cleavage, are shown as a function of irradiation and/or test temperature in Fig. 16. The cleavage fracture stress is reached before general yielding in most of the beryllium specimens tested at the lower temperatures as shown schematically in Fig. 17; the radiation damage enhances the shear stresses for basic and prismatic slip without significantly affecting the cleavage fracture stress so that the temperature at which some plasticity is restored is increased relative to that for the unirradiated beryllium. Furthermore, the data plotted in Fig. 18 show that the cleavage fracture stresses are proportional to $d^{-\frac{1}{2}}$, in accordance with the Hall - Petch relationship [equation (3)].

36. Recovery of the strengths and, to a limited extent, the ductilities occurs at the higher irradiation / test temperatures as a consequence of the annealing and annihilation of the point defects and clusters and coarsening of the intragranular helium bubbles. However, the ductilities may be further reduced at the high temperatures as the helium bubbles nucleated on the grain boundaries orthogonal to the applied tensile stress can grow continuously by the grain boundary vacancy diffusion process if the stress exceeds a critical value, assuming ideal gas behaviour, given by [41]:

$$\sigma_{crit} = 0.76\gamma/r_0 \quad (14)$$

where r_0 is the initial radius of the grain boundary gas bubble.

It follows, again assuming $\gamma = 1\ N.m^{-1}$, that helium bubbles with radii equal to or in excess of 7.6 nm lying on transverse grain boundaries will grow by this process at stresses of ≥ 100 MPa. The bubbles nucleate cavities which enlarge and coalesce to form cracks, resulting in premature intergranular failure with low ductilities in beryllium irradiated and tested at temperatures sufficiently high for the vacancies to be mobile. It is the fracture and not the deformation mode which is modified in this high temperature irradiation (helium) embrittlement process so that the ultimate tensile and proof stresses are not, in general, significantly affected.

Fracture Toughness

37. The fracture toughness tests were conducted on plain sided compact - tension (C-T) disc specimens (W = 16 mm, B = 8 mm) [1]. The specimens were machined in the L-R orientation from the cylindrical VHP billets, that is, with the diameter parallel to the direction of pressing (L) and crack propagation in the radial (R) direction, and in the L-T orientation, where T is the thickness (minor dimension), from the rectangular HIP billets.

38. Preliminary fracture toughness tests on the C-T specimens of the S - 200F. HIP beryllium were performed at the South West Research Institute, San Antonio, Texas [42]; the results on two disc specimens satisfied the ASTM E - 399 validity requirements for plane strain LEFM tests on beryllium [43][44] and yielded consistent K_{Ic} values of about 9.90 MPa$\sqrt{m}$ [1].

39. All the C-T specimens in the BSBE Programme were fatigue pre - cracked according to the ASTM E - 399 standard [45]. It was established, in agreement with previous experience with beryllium [20][46], that the cracks, once initiated, propagated rapidly to failure unless a negative R value (ratio of minimum to maximum stresses) was used. Thus, a typical R value of about - 2.5 was employed and the maximum stress intensity factor (K_{max}) was maintained at ≤ 9 MPa$\sqrt{m}$ during the fatigue pre - cracking.

40. The load - displacement curves were linear for most of the unirradiated (reference) and thermally aged specimens tested at temperatures below about 250°C and for the majority of the irradiated (fatigue cracked before reactor exposure) samples. The fracture toughness, K_Q, was therefore determined according to the recommended LEFM procedure and the K_{Ic} values quoted if all the ASTM validity requirements [43][44] were satisfied. The load - displacement curves for the unirradiated specimens tested at ≥ 250 C and some of the irradiated samples tested at the higher temperatures were not linear. The fracture toughness was therefore evaluated according to the EPFM J - integral procedures [47]. The equivalent stress intensity factor K_{Jc} (= $\sqrt{JE / 1 - \nu^2}$) represents the fracture toughness when the fracture occurs in an unstable manner. However, it was difficult to define the point at which the crack initiated when the fracture occurred in a stable manner and the fracture toughness ($K_{Jc\,Fmax}$) was therefore determined at the maximum load.

41. The fracture toughness values for the S-65 (HIP and VHP) and S-200F (HIP and VHP) beryllium grades are plotted as a function of ageing, irradiation and/or test temperature in Figs. C1 and C2 in Appendix C; the influences of (a) ageing at 185 and 230°C and irradiation

at 200 and 235 C on the fracture toughness at ambient temperature, and (b) ageing at temperatures in the range 310 to 605 C and irradiation at 350, 435, 600 and 610 C on the fracture toughness at 250 C are shown in Fig. D1 in Appendix D.* The principal observations may be summarised as follows:

(i) The fracture toughness of the unirradiated (reference) beryllium grades increases with increasing test temperature up to a maximum at 455 C (S-65. VHP, S-200F. HIP and VHP) or 540 C (S-65. HIP) and then decreases at higher temperatures.

(ii) Thermal ageing at 455 C increases the fracture toughness of all the beryllium grades in the tests at this temperature; the prior ageing has relatively little effect at the other test temperatures except in the case of the S-200F. VHP beryllium where there is a reduction of the fracture toughness at the lower temperatures and an increase at about 600 C.

(iii) The fracture toughness at all test temperatures above ambient are significantly higher for the unirradiated (reference) S-65 beryllium in the VHP than in the HIP condition. The differences between two S-200F grades are less pronounced but the fracture toughness of the VHP material appears to be superior to that of the HIP at test temperatures of $\leq 350^{\circ}$C, is approximately the same at 455 C and is inferior at 540 and about 600°C. However, prior thermal ageing generally reduces the differences between the respective VHP and HIP grades.

(iv) The fracture toughness values of the S-65 and S-200F HIP grades in the unirradiated condition are comparable at all temperatures whereas they are vastly superior for the S-65. VHP compared to the S-200F. VHP beryllium over the whole test temperature range.

(v) Irradiation has an extremely detrimental influence on the fracture toughness of the beryllium grades at all irradiation/test temperatures, the irradiation - induced reductions being most pronounced at about 450 C; the toughness at temperatures of $\leq 230^{\circ}$C is often reduced to below the fatigue pre - cracking level of about 9 MPa$\sqrt{m}$ but some recovery is evident above about 435 C.

(vi) The differences in the fracture toughness of the respective beryllium grades in the irradiated condition are relatively small, with the exception that the S-200F. HIP beryllium is much more ductile at irradiation/test temperatures of 435°C and, to a lesser extent, 600 C.

(vii) Ageing at 185 and 230 C has little effect but irradiation at 200 and 230 C reduces the ambient temperature fracture toughness of all the beryllium grades (Fig. D1 in Appendix D). The ageing produces small increases in the 250 C fracture toughness of the S-65. HIP and S-200F. HIP materials but substantially raises that of the S-65. VHP and, possibly, that of the S-200F. VHP beryllium. The irradiation significantly decreases the fracture toughness in the tests at 250°C but the values tend to be higher than those of the irradiated samples tested at room temperature; the fracture toughness values at 250°C also show moderate recovery for all the materials, except those of the S-200F. HIP grade, following irradiation at 600 and 610°C.

42. The ambient temperature fracture toughness values of about 11 MPa$\sqrt{m}$ for the

* The quoted fracture toughness values may be adjusted when the results of continuing studies at SCK / CEN, Mol, aimed at defining the ductile crack initiation point more precisely, are available.

unirradiated beryllium grades are in reasonably good agreement with those reported in the literature. Thus, the K_Q (K_{1c}) values for HIP and VHP beryllium produced using different powder production techniques and with BeO contents of 0.37 - 1.7% and grain sizes of 7.2 - 13.2 μm range from 9.1 to 10.6 MPa$\sqrt{m}$ for specimens machined in the L-T orientation and 9.2 to 13.6 MPa$\sqrt{m}$ for the T-L orientation [20]. Other plane strain fracture toughness tests give K_Q values increasing from approximately 8 to 16 MPa$\sqrt{m}$ with increasing test temperature from - 200 to + 270 C for S-200E beryllium [48] and about 9 MPa$\sqrt{m}$ at room temperature and 300 C for high strength beryllium [49].

43. The fracture toughness values decrease progressively with increasing tensile proof and/or ultimate stresses for the unirradiated (reference) beryllium grades at test temperatures in the range 185 to 455 C [Fig. 19 (a) and (c)] and for the thermally aged materials at 185, 230 and 310°C [Fig. 19 (b)]. These inverse dependences of the fracture toughness on the tensile strengths imply that an increased grain size within the range 6.6 to 8.4 μm and reduced lattice hardening enhance the toughness at the low and intermediate test temperatures investigated in the BSBE Programme. However, the fracture toughness of the beryllium in the unirradiated (reference) condition at test temperatures of 540 and 605°C and of the thermally aged grades at 455°C increase with increasing reduction of area values in the tensile tests at the same temperature [Fig. 19 (d)]]. It follows that the fracture toughness of the beryllium at these higher test temperatures are reduced with increasing concentrations of BeO, other coarse inclusions and precipitates, and / or impurity elements; these features may restrict the non - uniform deformation in the tensile tests by promoting intragranular microvoid nucleation and coalescence and intergranular cavitation failures. It is noted, however, that an increase in purity is claimed to have only a limited beneficial effect on the ambient temperature fracture toughness of hot pressed beryllium stock [21][40].

44. Although the irradiation and test temperatures, displacement doses and helium concentrations are not precisely the same, the data in Fig. 20 illustrate that the tensile proof stresses and reductions of area and the fracture toughness exhibit similar dependences on the BeO (and / or impurity) content of the beryllium. The superior toughness of the S-200F. HIP grade and the inferior toughness of the S-200F. VHP material in the post - irradiation tensile and fracture toughness tests at 485 and 435 C respectively are again apparent.

45. There is a paucity of data on the effects of irradiation on the fracture toughness of beryllium. The only publication records a reduction of 60 and 68% in the fracture toughness of hot pressed nuclear grade and porous beryllium (initial values of 12 and 13.1 MPa$\sqrt{m}$) respectively following irradiation at 66 C to 3.5 - 5.0 x 10^{25} n.m^{-2} (> 1 MeV) [50].

Conclusions

46. The main conclusions of this assessment are as follows:

(i) The tensile proof and ultimate strengths of the HIP and VHP S-65 and S-200F beryllium grades in the unirradiated (reference) and thermally aged conditions decrease progressively with increasing temperature from ambient to 605°C and, at a given temperature, increase with the inverse square root of the grain size (d) in accordance with the Hall - Petch relationship.

(ii) The results of an analysis of the tensile stress - strain curves for the S-200F. HIP beryllium suggest that the yield stresses are determined principally by the impurities, precipitates, etc. which increase the lattice friction stress opposing the movement of free dislocations. However, the tensile strengths do not show a consistent dependence on the BeO content of the beryllium within the range of 0.5 to 1.2%.

(iii) The tensile ductilities of the unirradiated beryllium increase to a maximum at 230 - 310°C and then decrease at higher temperatures; thermal ageing increases the non - uniform ductilities of the S-200F grades in tests at 455 - 600°C, possibly as a result of the redistribution and/ or precipitation of the impurity atoms.

(iv) The non - uniform tensile ductilities generally decrease with increasing BeO and/or impurity content at test temperatures in the range 310 to 605°C; however, there is no strong correlation between the ductility and grain size although other observations suggest a total elongation - $d^{-\frac{1}{2}}$ dependency.

(v) The present observations on the unirradiated (reference) and aged tensile tested beryllium samples are consistent with a change in primary fracture mode from brittle transgranular cleavage to ductile (dimple) fibrous and, possibly, intergranular with increasing temperature.

(vi) Irradiation produces marked embrittlement at irradiation/test temperatures of 185, 235 and 310°C but there is some recovery of the ductility at the higher temperatures of 485, 540 and 600°C. The enhanced tendency to cleavage fracture at the lower temperatures is associated with a larger irradiation - induced increase in the yield than in the fracture stress; the cleavage fracture stress is dependent on $d^{-\frac{1}{2}}$, again in agreement with the Hall - Petch relation. The irradiated samples show intergranular and/or ductile dimple fracture at the higher temperatures; the intergranular cracking probably results from the stress - induced growth and coalescence of adjacent grain boundary helium bubbles.

(vii) There is little to chose between the various beryllium grades in respect of their resistance to radiation damage at the lower irradiation/tensile test temperatures ($\leq$ 310°C) as they are all brittle; however, the S-65 VHP and, in particular, the S-200F HIP grades are more ductile at 435 to 600°C.

(viii) The fracture toughness values for the unirradiated (reference) and aged beryllium are broadly in agreement with the tensile data in that they increase up to a maximum at intermediate temperatures and decrease again at higher temperatures. The fracture toughness decreases with increasing tensile strengths at the low and intermediate temperatures and increases with increasing tensile reductions of area at the higher test temperatures.

(ix) Irradiation markedly reduces the fracture toughness at all temperatures but there is some recovery at an irradiation/test temperature of about 600°C; there are only small differences in the fracture toughness of the irradiated beryllium grades although the S-200F. HIP material is tougher at the irradiation/test temperature of 435°C and to a lesser extent at 600°C. Limited data suggest that there is a reasonable correlation between the fracture toughness and the tensile proof stress and / or reduction of area values for the irradiated beryllium.

<u>Limitations of the Existing Data and Future Requirements</u>

47. The BSBE Programme was initiated to provide data in support of the proposed applicat-

ion of beryllium as a neutron multiplier in a DEMO fusion power reactor blanket design based on the use of a solid lithium ceramic for the tritium breeding and helium as the coolant. However, this initial concept, in which the beryllium is in the form of plates, has now been abandoned in favour of a design utilising small beryllium pebbles and, as a consequence, many of the original material and design data requirements are no longer applicable. Nevertheless, it is considered that the following additional investigations would be necessary to validate some of the conclusions of the present assessment with respect to the factors determining the tensile and fracture toughness properties and behaviour of the unirradiated and irradiated beryllium, to advance our understanding of the effects of irradiation and, thereby, enable further progress to be made in establishing material composition - structure - property relationships:

(i) Detailed analytical TEM examinations of (a) the structure (BeO particles, precipitates, dislocations, etc.) of the unirradiated (reference) and thermally aged beryllium, and (b) the radiation damage structure (point defect clusters, dislocation loops, inter - and intra - granular helium bubble sizes and densities, interfacial segregation and precipitation) as a function of beryllium grade and irradiation temperature, displacement damage and helium concentration.

(ii) Further SEM examinations of the existing specimens to elucidate the effect of temperature on crack nucleation and propagation modes for both the unirradiated and irradiated beryllium samples.

(iii) Additional irradiations of specimens, ideally to a number of equivalent displacement damage and helium concentrations at several irradiation temperatures in the range up to 600°C followed by tensile and fracture toughness testing at the same temperatures, so as to allow a more precise evaluation of the effects of the irradiation variables to be made for a limited number of modern beryllium grades.

(iv) Development of an isotropic commercial beryllium product with a sub - micron grain size and low BeO and elemental impurity contents in an attempt to increase the cleavage fracture stress and the ductility and toughness at the lower temperatures. (It is probable that this objective can never be fully realised because of the inherent characteristics of the beryllium crystals [20]).

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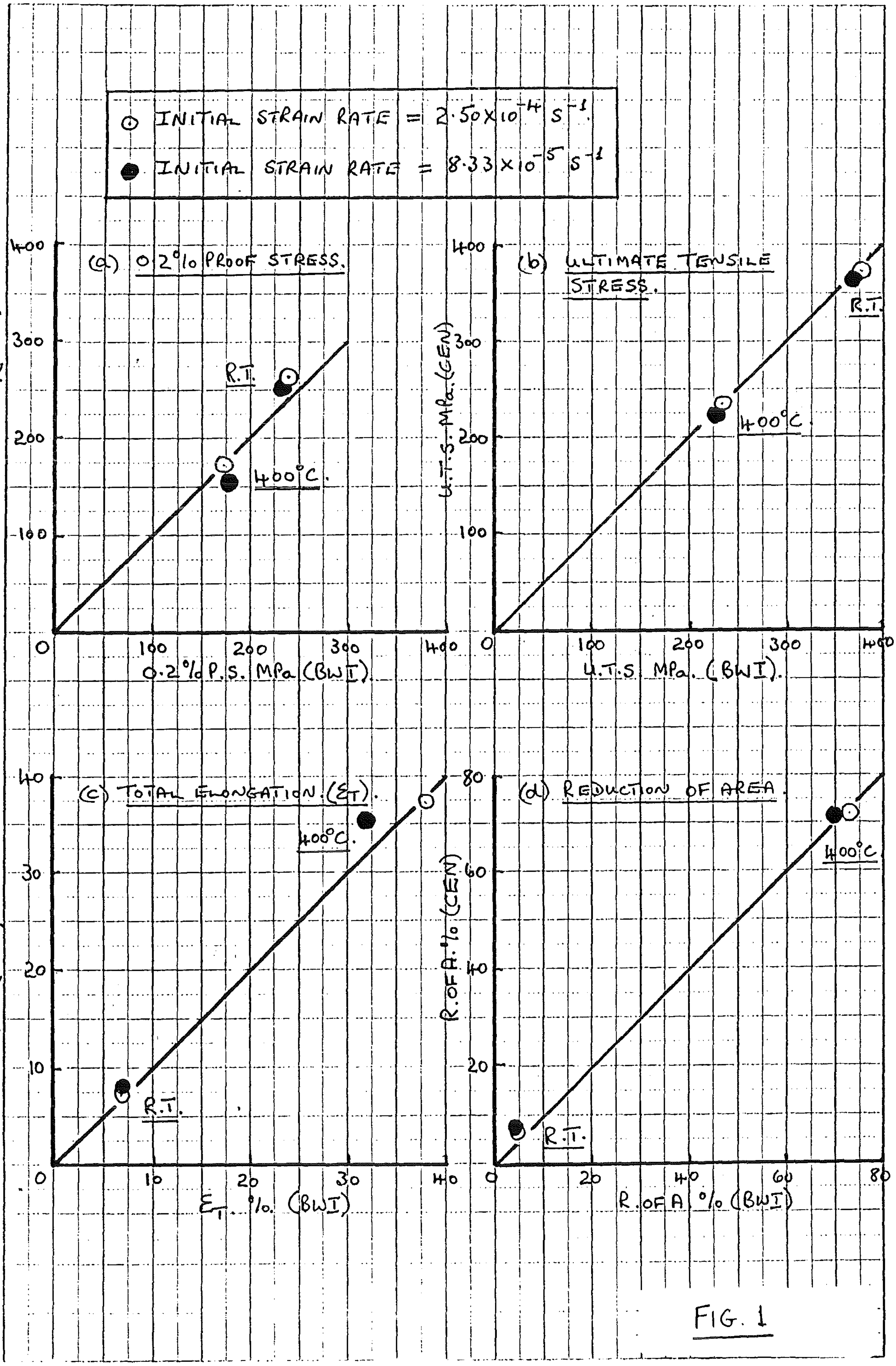
49. M.W. Perra and I. Finnie, J. Mater. Sci., 12 (1977) 1519.
50. J.M. Beeston, ASTM Special Tech. Pubn. 683, 1979, p. 309.

<u>Figure Captions</u>

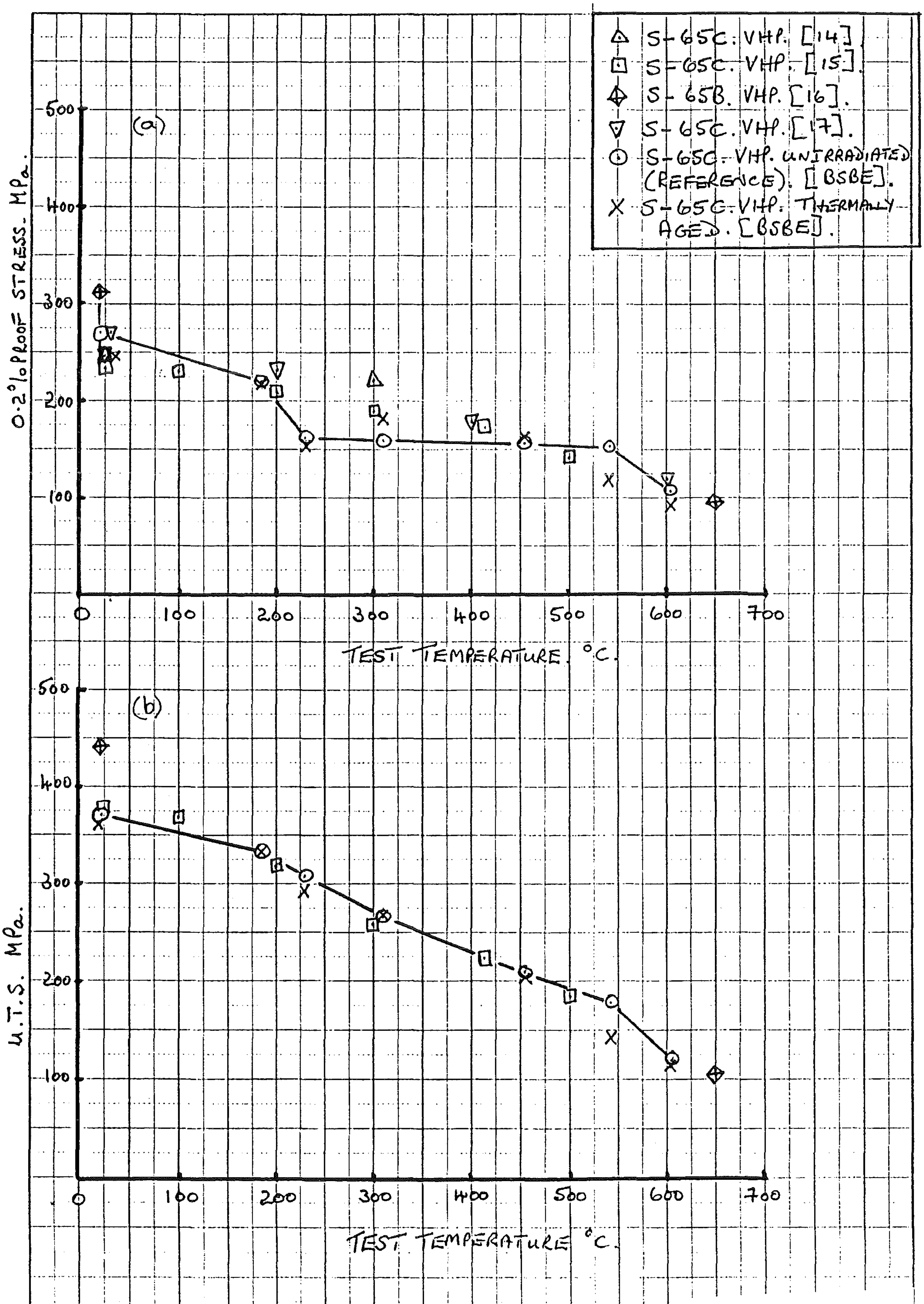
<u>Fig</u>.

1. Comparison of the tensile properties of S-65C. VHP beryllium at ambient temperature and 400°C and initial strain rates of 2.50 x 10^{-4} and 8.33 x 10^{-5} s^{-1} measured using the CEN (BSBE) and BWI type specimens.
2. Comparison of the BSBE and published data on the ambient and elevated temperature tensile properties of S-65. VHP beryllium.
3. Comparison of the BSBE and BWI data on the tensile proof strengths and total elongations at ambient and elevated temperatures of S-200F. VHP beryllium.
4. Hexagonal close packed (hcp) crystal showing basal and prism planes.
5. Effect of temperature on the critical stresses for slip, twinning and fracture in beryllium [19].
6. Illustration of slip and cracking at the tips of slip bands.
7. Illustration of typical tensile load - displacement curves for unirradiated and irradiated beryllium.
8. Tensile load - displacement curve at ambient temperature for unirradiated (reference) S-200F. HIP beryllium [5].
9. Tensile load - displacement curve at 310 C for unirradiated (reference) S-200F. HIP beryllium [5].
10. Tensile load - displacement curve at 605 C for unirradiated (reference) S-200F. HIP beryllium [5].
11. Correlation of the tensile 0.2% proof stresses at test temperatures in the range ambient to 605°C with the grain sizes of the respective beryllium grades.
12. Correlation of the ultimate tensile strengths at test temperatures in the range ambient to 605°C with the grain sizes of the respective beryllium grades.
13. Illustration of the method for determining σ_i and k_y by extrapolation of the work hardening portion of the tensile stress - strain curve; the dotted line is determined from a plot of log true stress versus log true strain.
14. Effect of BeO content on the tensile proof stresses and ductilities of the beryllium in the unirradiated (reference) condition at temperatures in the range ambient to 605°C.
15. Effect of BeO content on the tensile reduction of area values of the beryllium in the unirradiated (reference) and thermally aged conditions at temperatures in the range 310 to 605°C.
16. Effects of irradiation and/or test temperature on the tensile 0.2% proof and cleavage fracture stresses of the beryllium grades.
17. Schematic illustration of the effects of irradiation on the critical stresses for slip and fracture in beryllium.
18. Effect of grain size on the cleavage fracture stress for the irradiated beryllium.
19. Correlations of the fracture toughness with the tensile proof and ultimate strengths and reductions of area at elevated temperatures for the unirradiated (reference) and thermally aged beryllium grades.
20. Dependences of the tensile proof stresses and reductions of area and fracture toughness on the BeO contents of the beryllium grades irradiated and tested at 485/435°C.



INITIAL STRAIN RATE = 2·50×10⁻⁴ s⁻¹
INITIAL STRAIN RATE = 8·33×10⁻⁵ s⁻¹
(a) 0.2% PROOF STRESS.
0.2% P.S. MPa. (CEN).
0.2% P.S. MPa (BWI).
(b) ULTIMATE TENSILE STRESS.
U.T.S. MPa. (CEN)
U.T.S. MPa. (BWI).
(c) TOTAL ELONGATION (ε_T).
ε_T % (CEN).
ε_T % (BWI)
(d) REDUCTION OF AREA.
R. OF A. % (CEN)
R. OF A. % (BWI)
R.T.
400°C.

FIG. 1



S-65C. VHP. [14].
S-65C. VHP. [15].
S-65B. VHP. [16].
S-65C. VHP. [17].
S-65C. VHP. UNIRRADIATED (REFERENCE). [BSBE].
S-65C. VHP. THERMALLY AGED. [BSBE].
(a)
0.2% PROOF STRESS. MPa.
500
400
300
200
100
0
100
200
300
400
500
600
700
TEST TEMPERATURE. °C.
(b)
U.T.S. MPa.
500
400
300
200
100
0
100
200
300
400
500
600
700
TEST TEMPERATURE °C.

FIG. 2

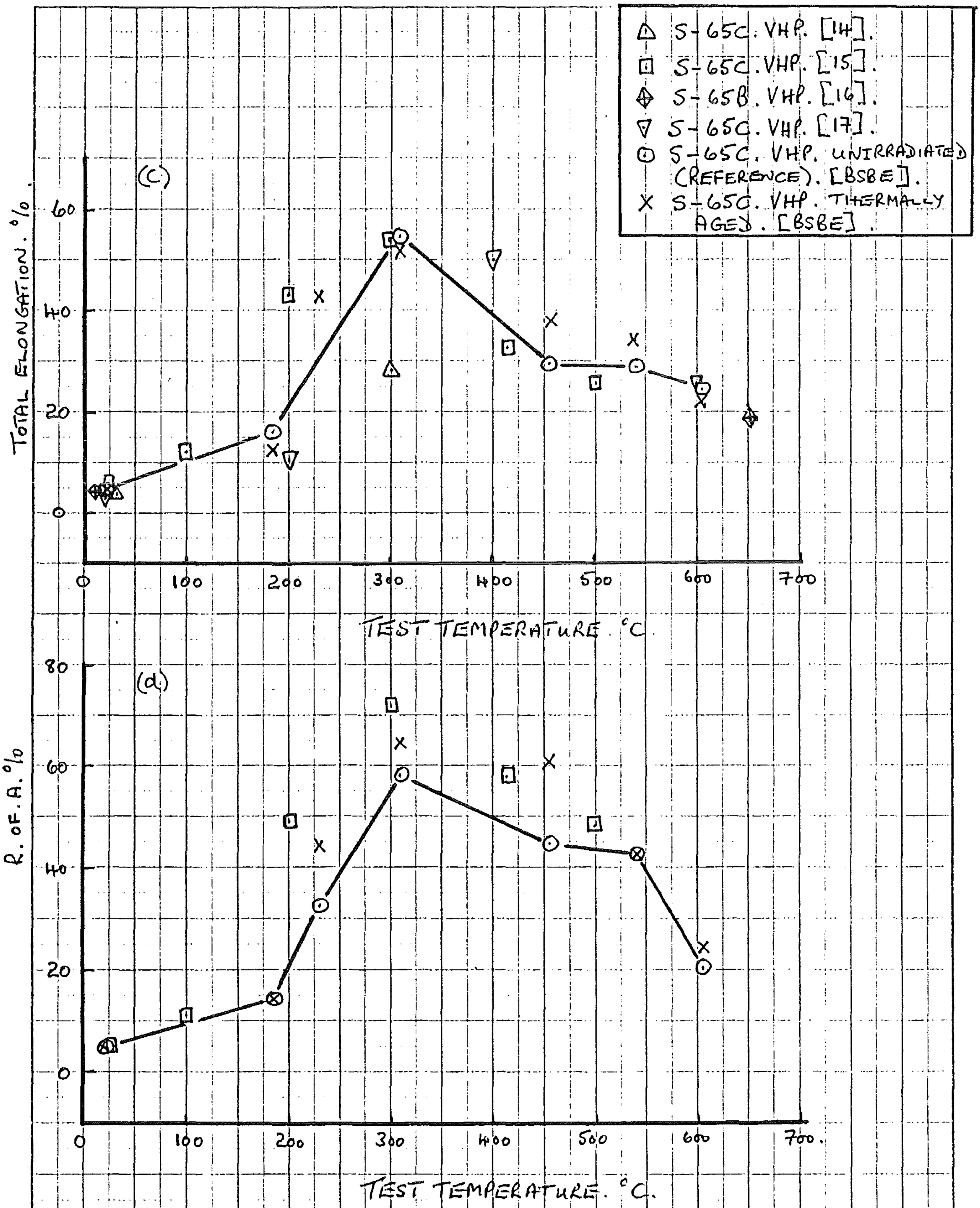

S-65C. VHP. [14].
S-65C. VHP. [15].
S-65B. VHP. [16].
S-65C. VHP. [17].
S-65C. VHP. UNIRRADIATED (REFERENCE). [BSBE].
S-65C. VHP. THERMALLY AGED. [BSBE].
(c)
TOTAL ELONGATION. %
0
20
40
60
0
100
200
300
400
500
600
700
TEST TEMPERATURE °C
(d)
R. OF. A. %
0
20
40
60
80
0
100
200
300
400
500
600
700.
TEST TEMPERATURE. °C.

FIG. 2 (CONT'D)

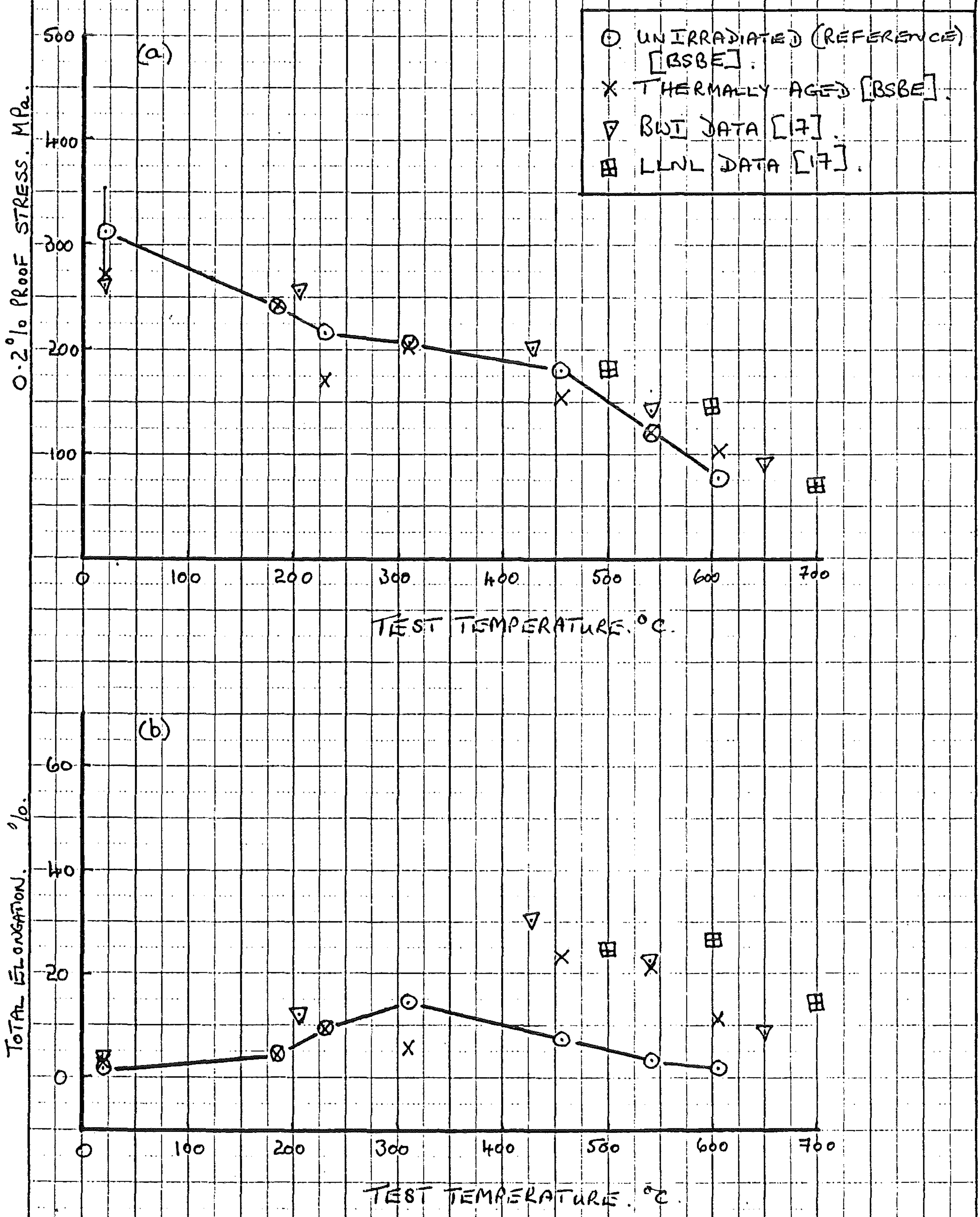

UNIRRADIATED (REFERENCE) [BSBE].
THERMALLY AGED [BSBE].
BWI DATA [17].
LLNL DATA [17].
(a)
0.2% PROOF STRESS. MPa.
500
400
300
200
100
0
100
200
300
400
500
600
700
TEST TEMPERATURE. °C.
(b)
TOTAL ELONGATION. %.
60
40
20
0
100
200
300
400
500
600
700
TEST TEMPERATURE. °C.

FIG. 3

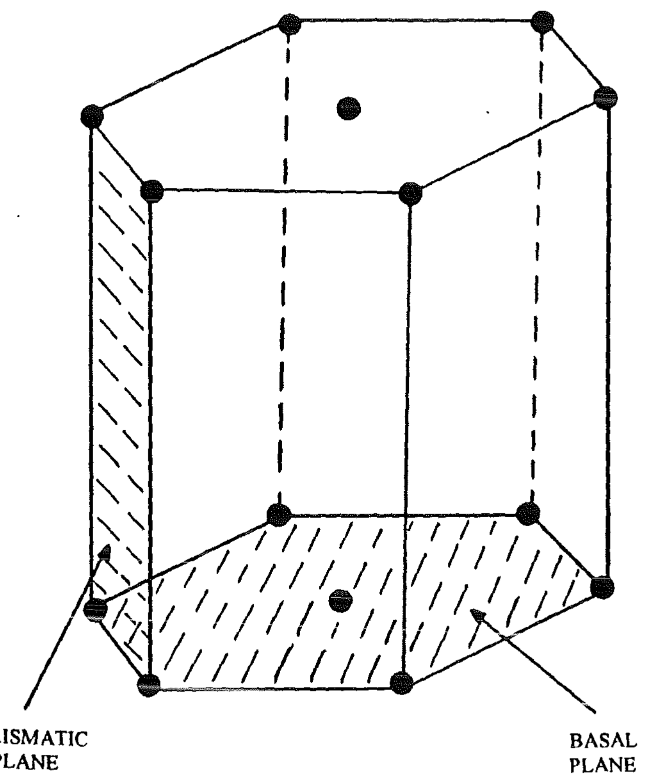


FIG. 4.

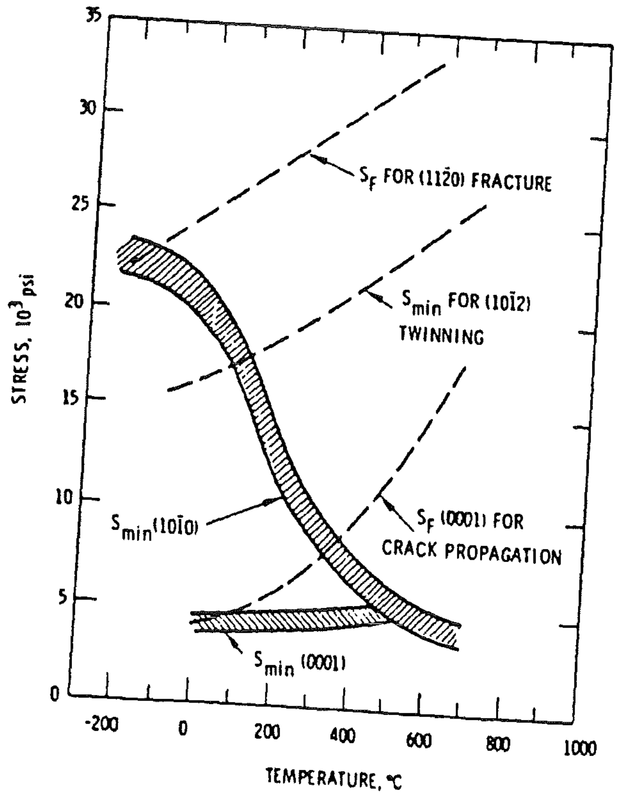


FIG. 5

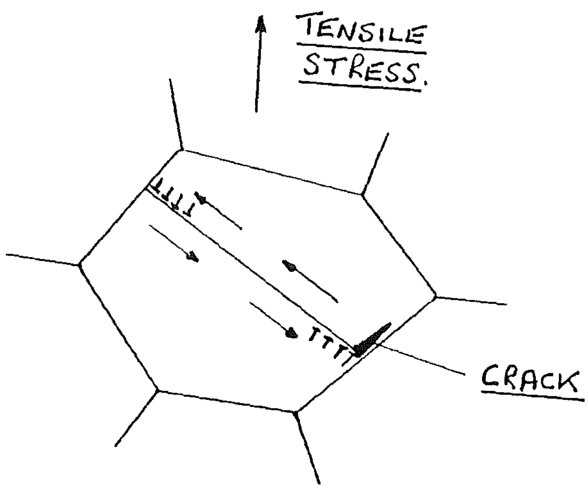


FIG. 6.

TENSILE (LOAD - DISPLACEMENT (STRESS - STRAIN) CURVES

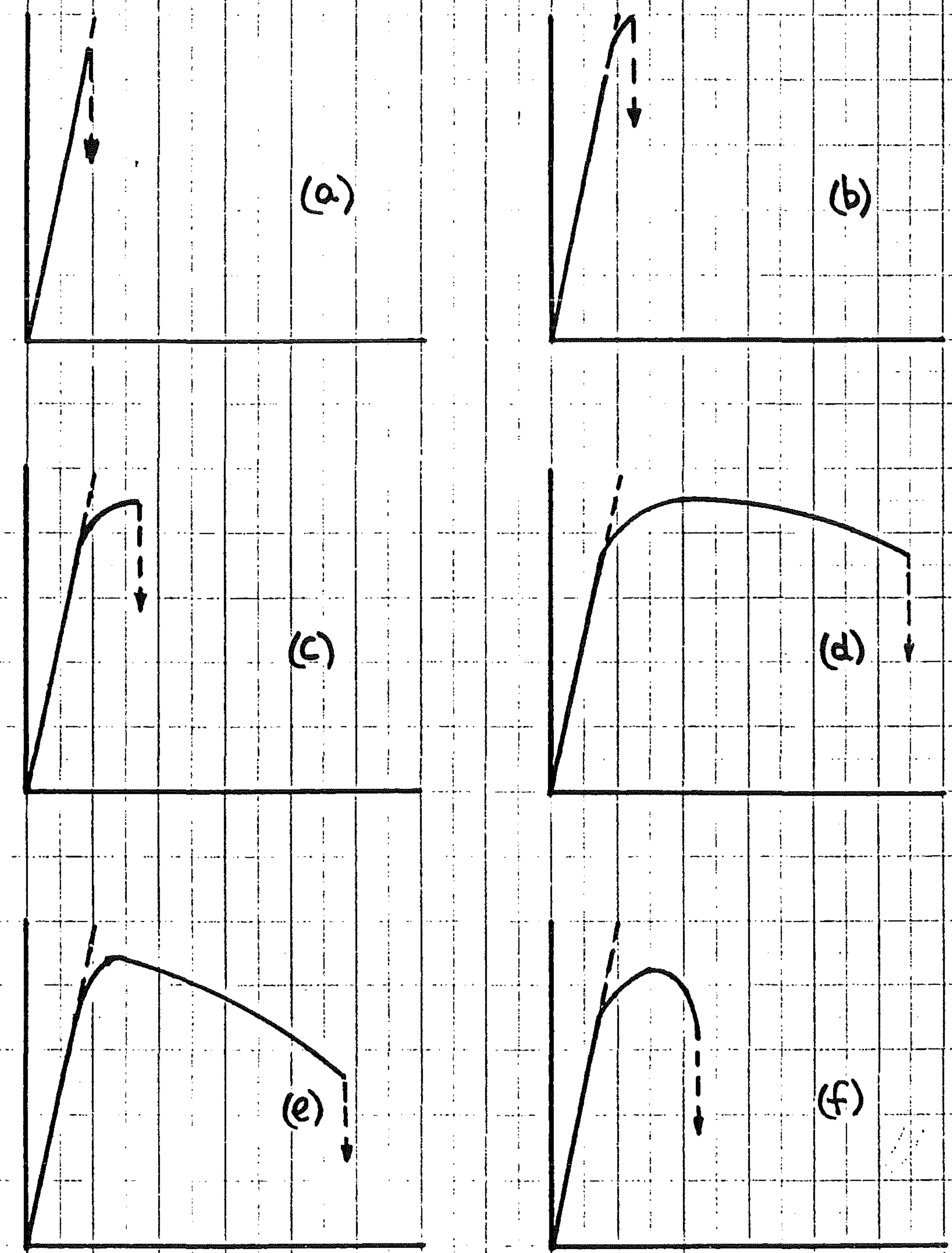


FIG. 7.

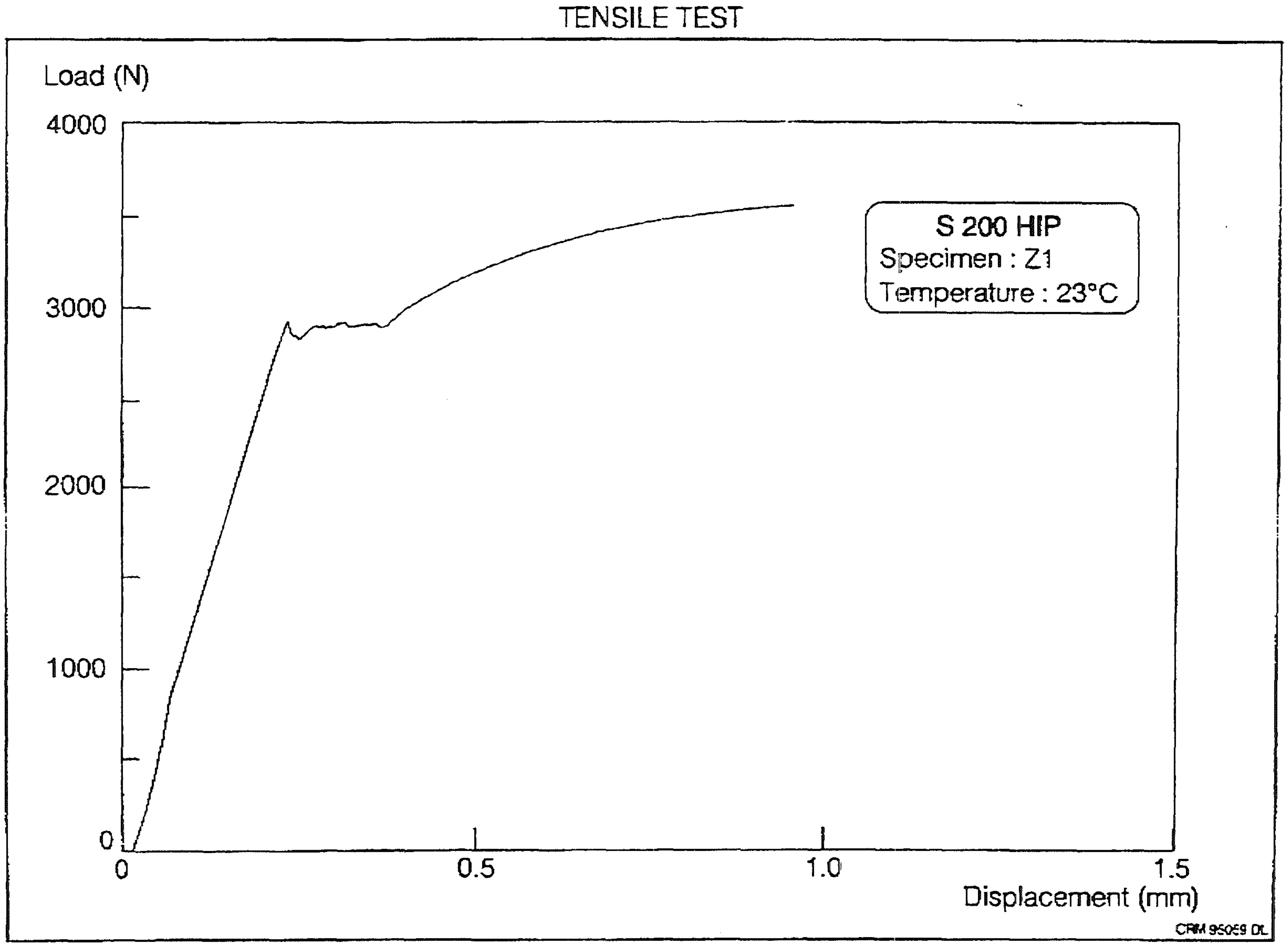
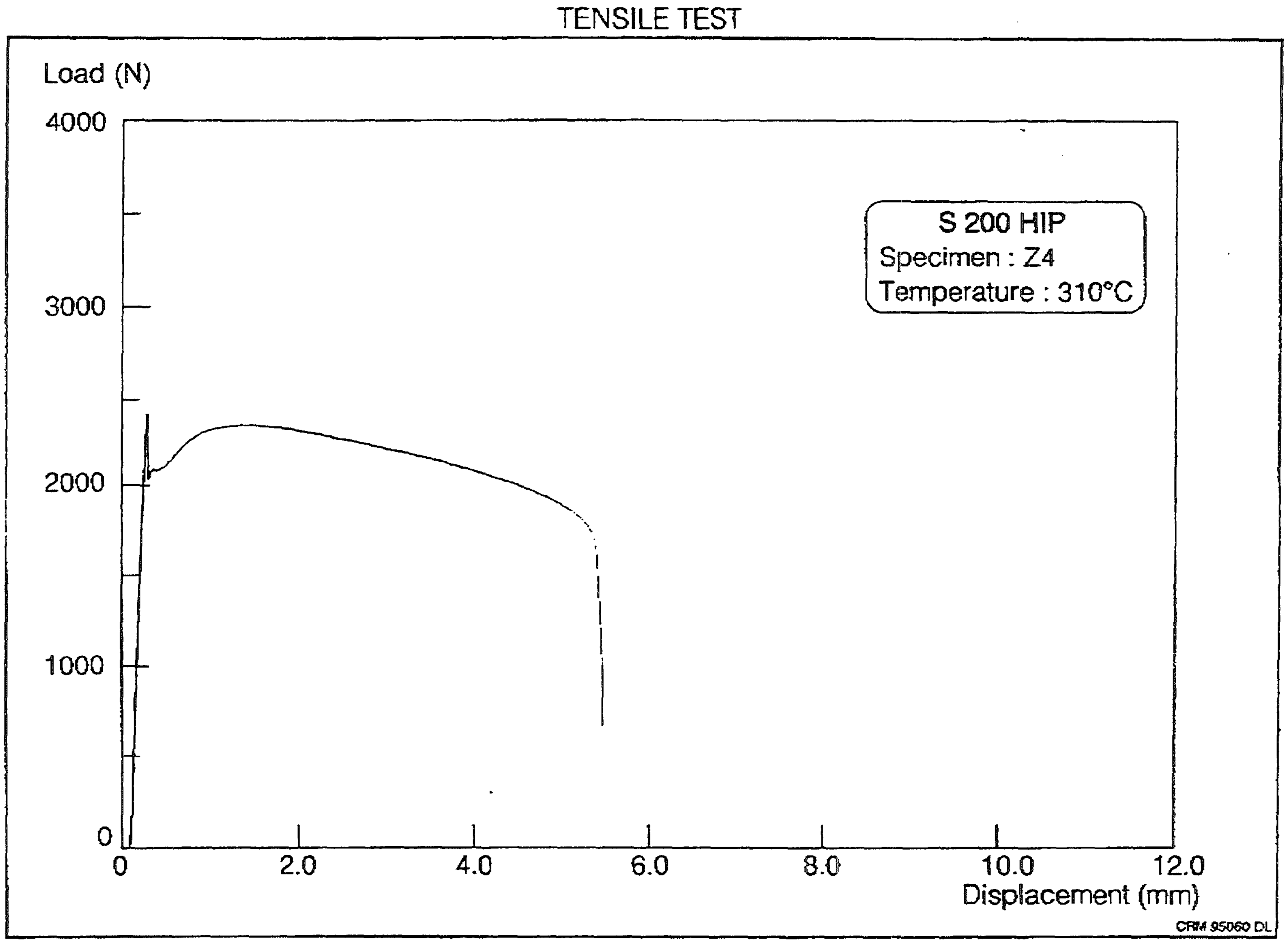
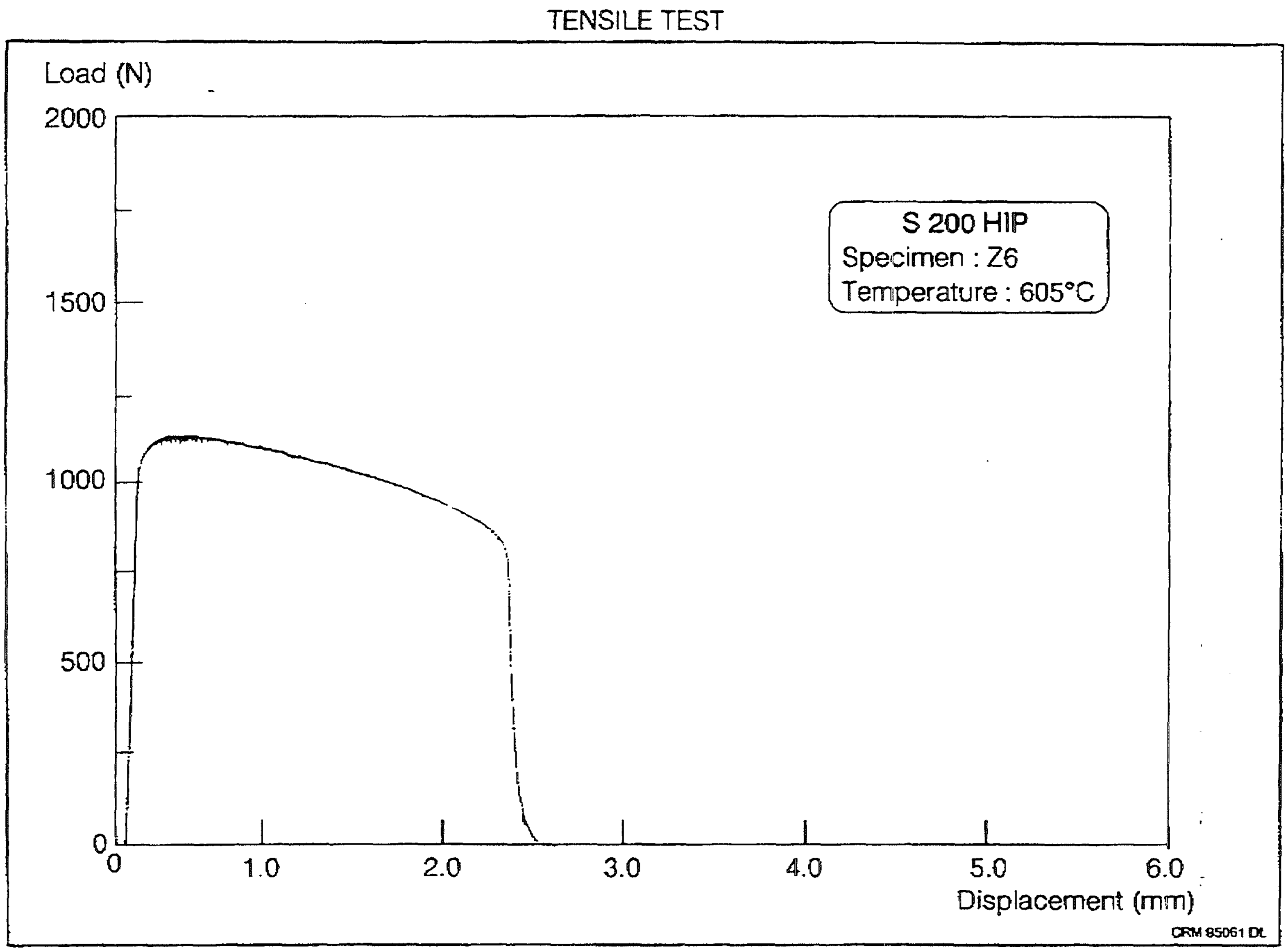

TENSILE TEST
Load (N)
4000
3000
2000
1000
0
0
0.5
1.0
1.5
Displacement (mm)
S 200 HIP
Specimen : Z1
Temperature : 23°C
CRM 95059 DL

FIG. 8.



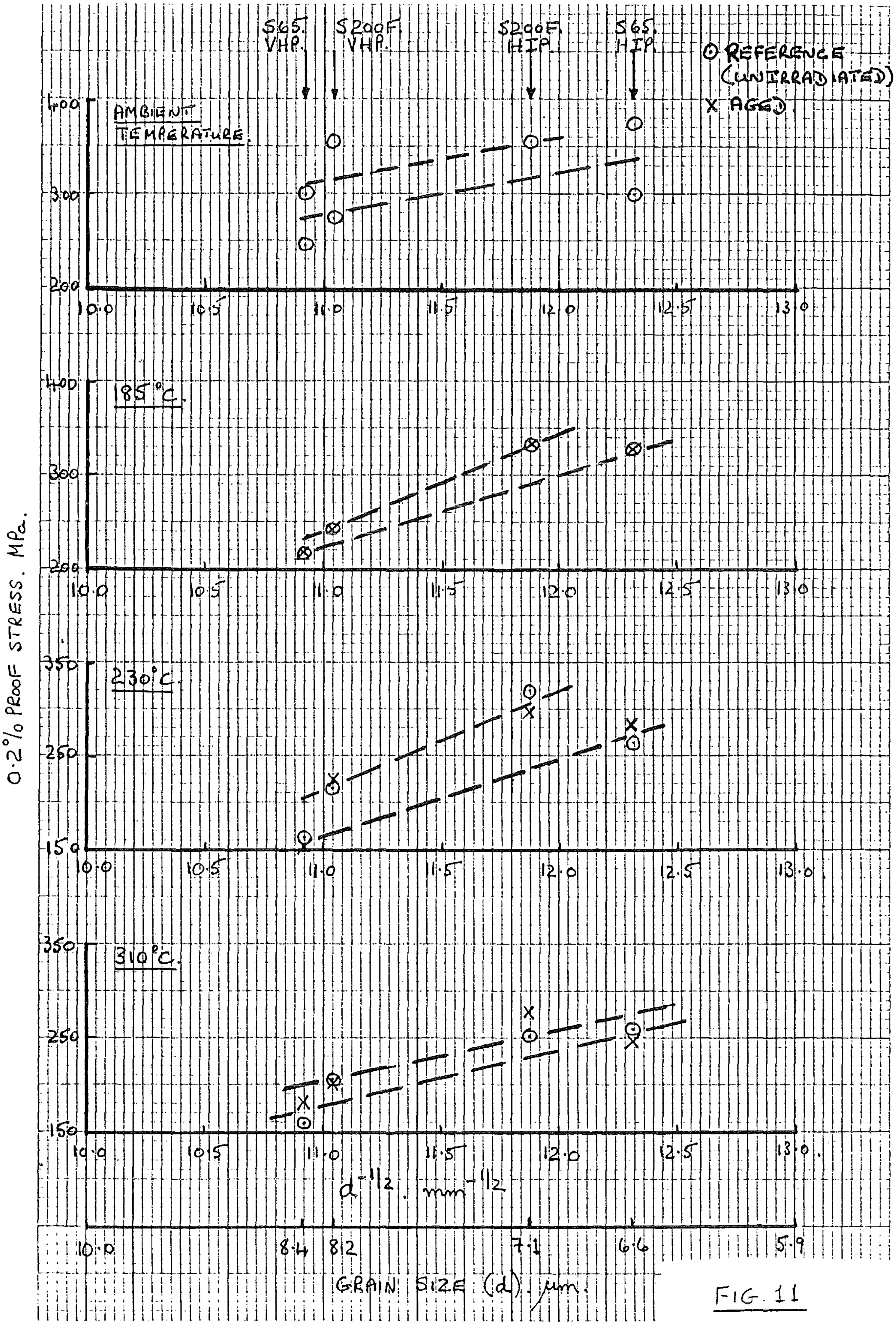
TENSILE TEST
Load (N)
4000
3000
2000
1000
0
0
2.0
4.0
6.0
8.0
10.0
12.0
Displacement (mm)
S 200 HIP
Specimen : Z4
Temperature : 310°C
CRM 95060 DL

FIG. 9



TENSILE TEST
Load (N)
2000
1500
1000
500
0
0
1.0
2.0
3.0
4.0
5.0
6.0
Displacement (mm)
S 200 HIP
Specimen : Z6
Temperature : 605°C
CRM 85061 DL

FIG. 10



S65. VHP.
S200F. VHP.
S200F. HIP.
S65. HIP.
⊙ REFERENCE (UNIRRADIATED)
X AGED
AMBIENT TEMPERATURE.
185°C.
230°C.
310°C.
0.2% PROOF STRESS. MPa.
400
300
200
350
250
150
10.0
10.5
11.0
11.5
12.0
12.5
13.0
$d^{-1/2}$. $mm^{-1/2}$
8.4
8.2
7.1
6.6
5.9
GRAIN SIZE (d). μm.

FIG. 11

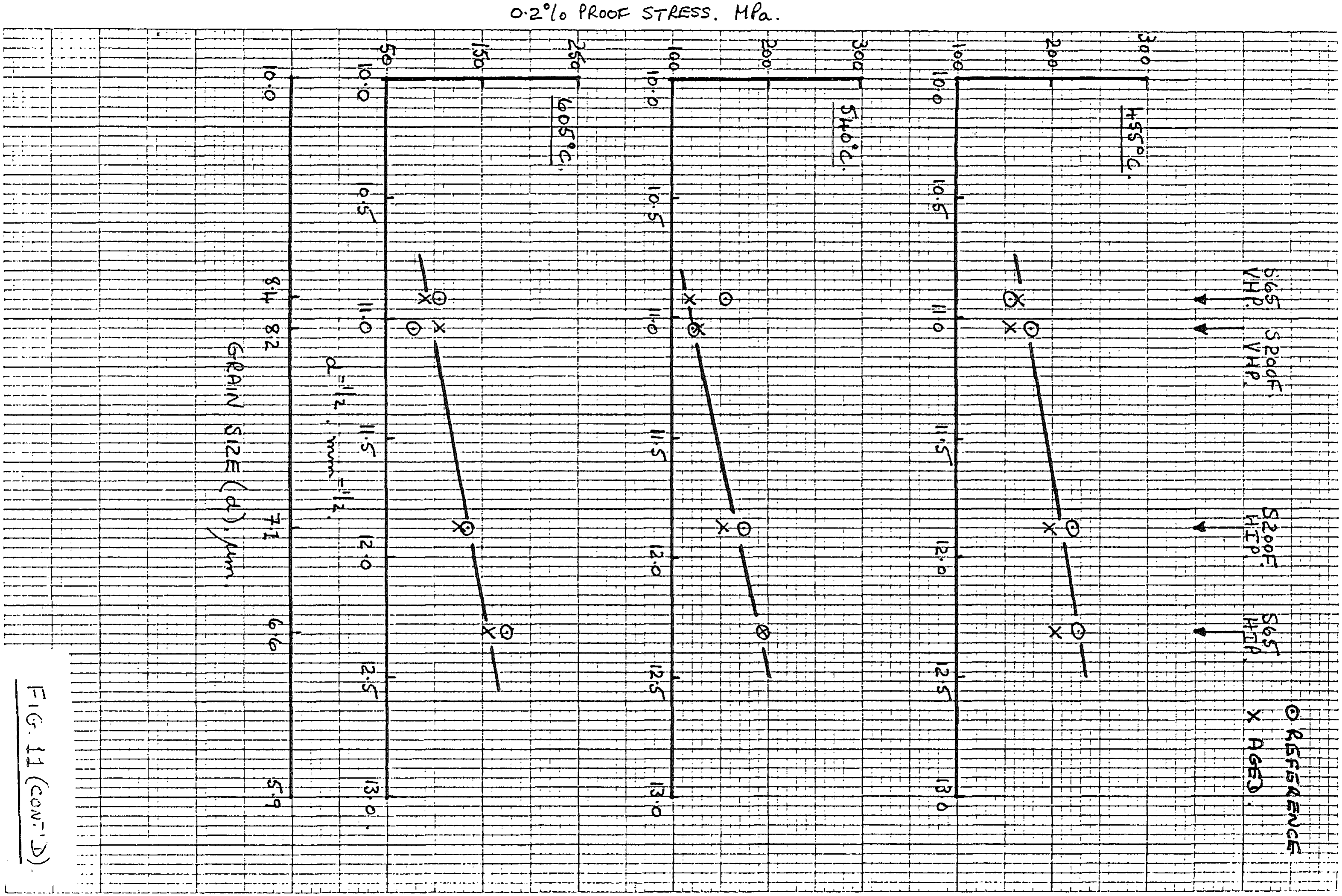
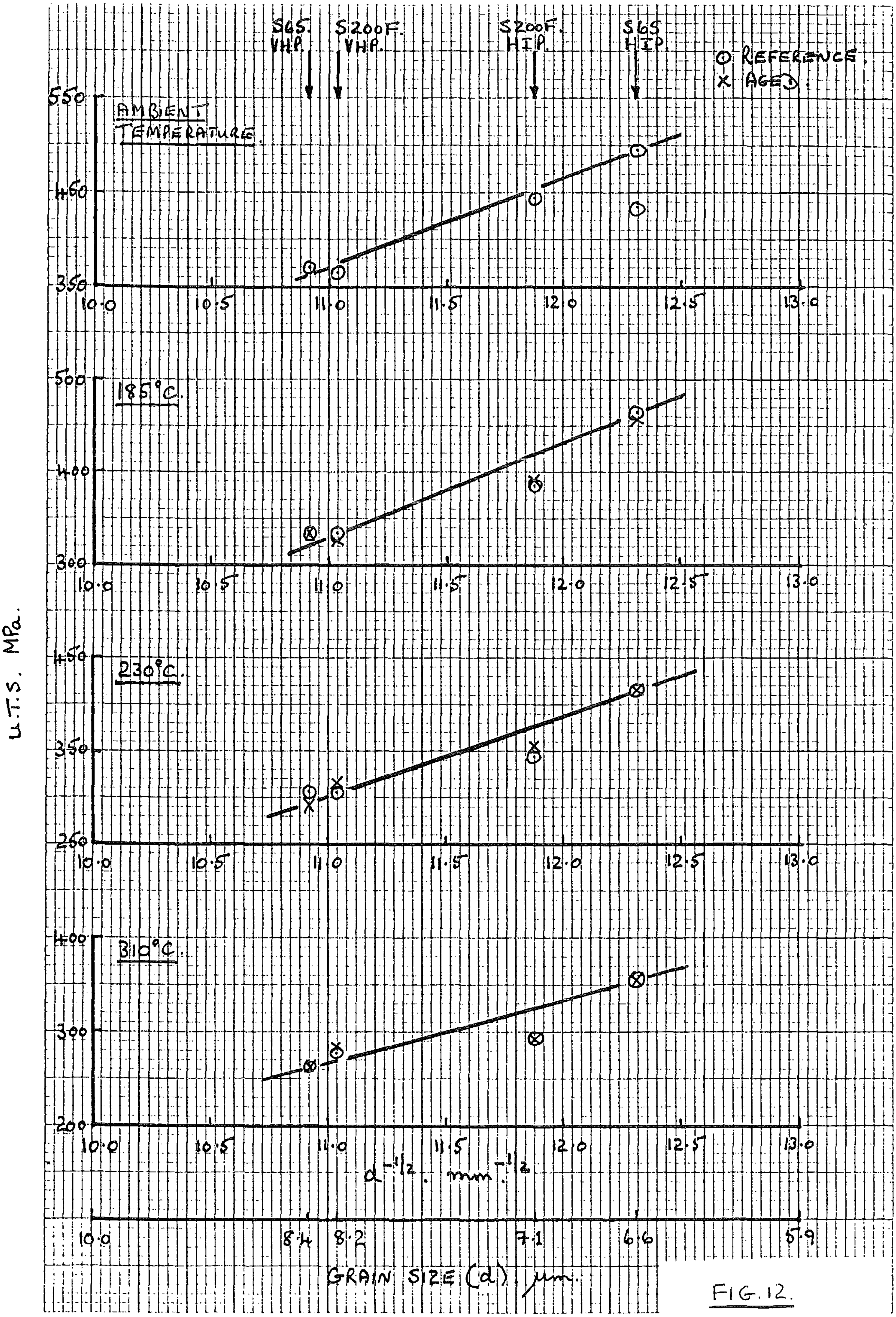
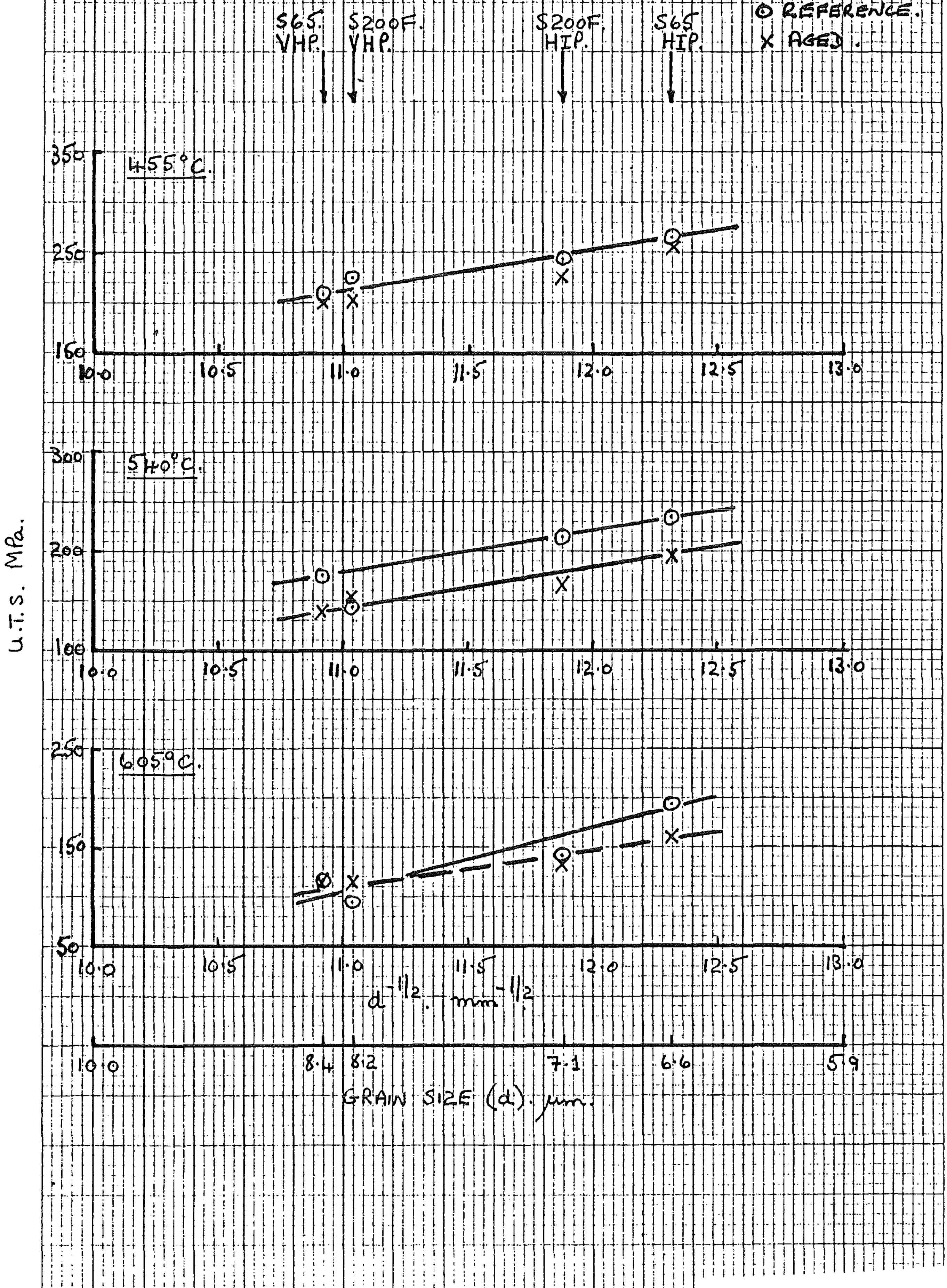

0·2% PROOF STRESS. MPa.
605°C.
540°C.
455°C.
50
150
250
100
200
300
100
200
300
10·0
10·5
11·0
11·5
12·0
12·5
13·0
d^-1/2 . mm^-1/2 .
10·0
8·4
8·2
7·1
6·6
5·9
GRAIN SIZE (d), μm.
S65 VHP.
S200F VHP.
S200F HIP.
S65 HIP.
⊙ REFERENCE
X AGED.

FIG. 11 (CONT'D).



S65. VHP.
S200F. VHP.
S200F. HIP.
S65 HIP.
⊙ REFERENCE.
× AGED.
AMBIENT TEMPERATURE.
185°C.
230°C.
310°C.
U.T.S. MPa.
550
450
350
500
400
300
450
350
250
400
300
200
10·0
10·5
11·0
11·5
12·0
12·5
13·0
$d^{-1/2}$. $mm^{-1/2}$
10·0
8·4
8·2
7·1
6·6
5·9
GRAIN SIZE (d) μm.

FIG. 12.



S65. VHP.
S200F. VHP.
S200F. HIP.
S65 HIP.
⊙ REFERENCE.
X AGED.
455°C.
350
250
160
10.0
10.5
11.0
11.5
12.0
12.5
13.0
540°C.
300
200
100
605°C.
250
150
50
U.T.S. MPa.
$d^{-1/2}$. $mm^{-1/2}$
10.0
8.4
8.2
7.1
6.6
5.9
GRAIN SIZE (d). μm.

FIG. 12 (CONT'D.)

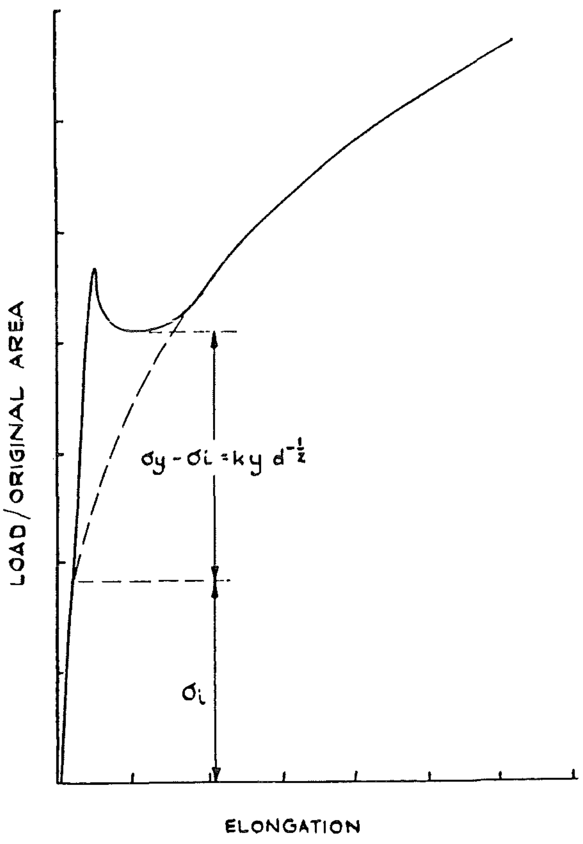

LOAD/ORIGINAL AREA
$\sigma_y - \sigma_i = k_y d^{-\frac{1}{2}}$
σ_i
ELONGATION

FIG. 13.

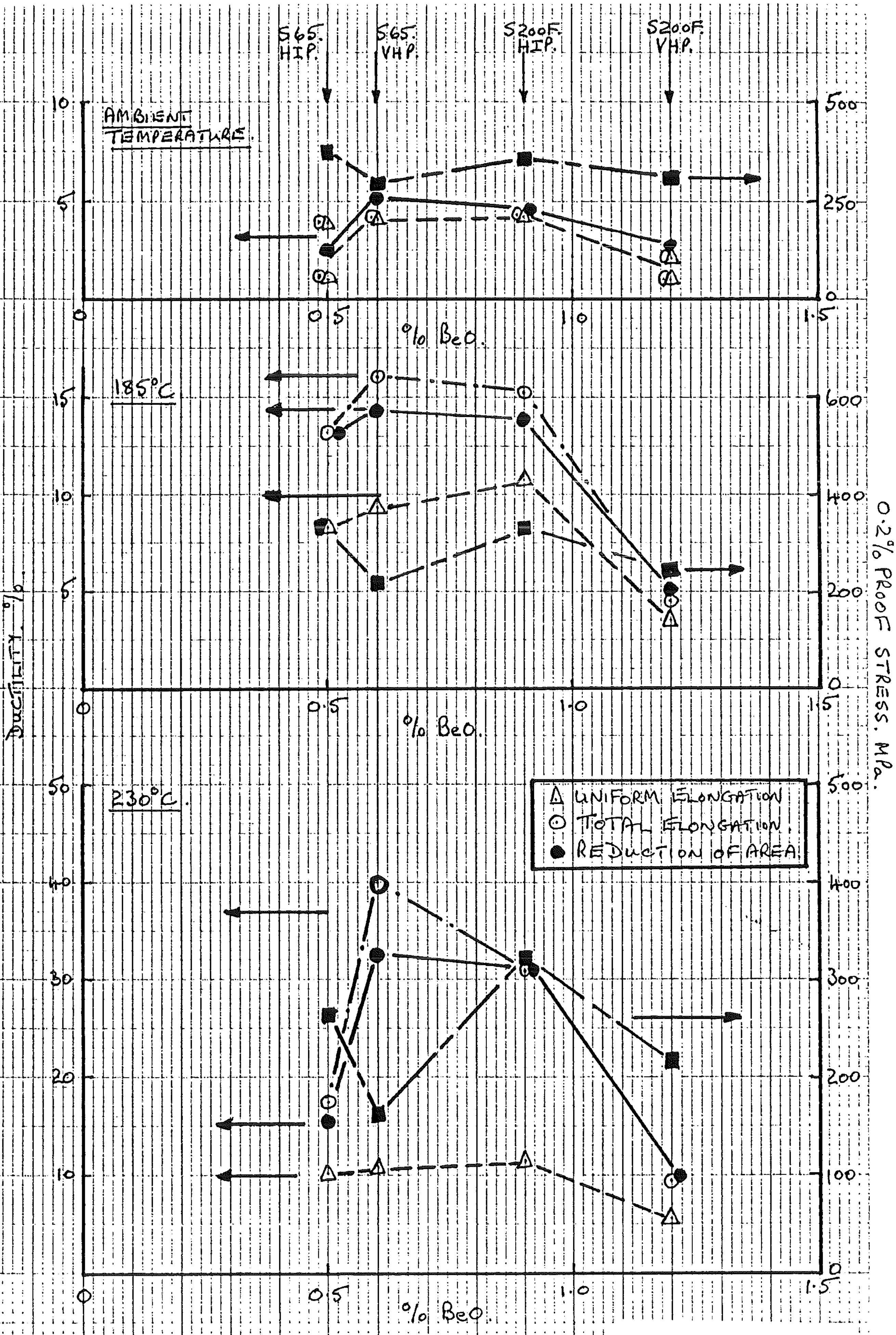

S65. HIP.
S65. VHP.
S200F. HIP.
S200F. VHP.
AMBIENT TEMPERATURE.
185°C.
230°C.
% BeO.
DUCTILITY. %
0.2% PROOF STRESS. MPa.
UNIFORM ELONGATION
TOTAL ELONGATION.
REDUCTION OF AREA.

FIG 14

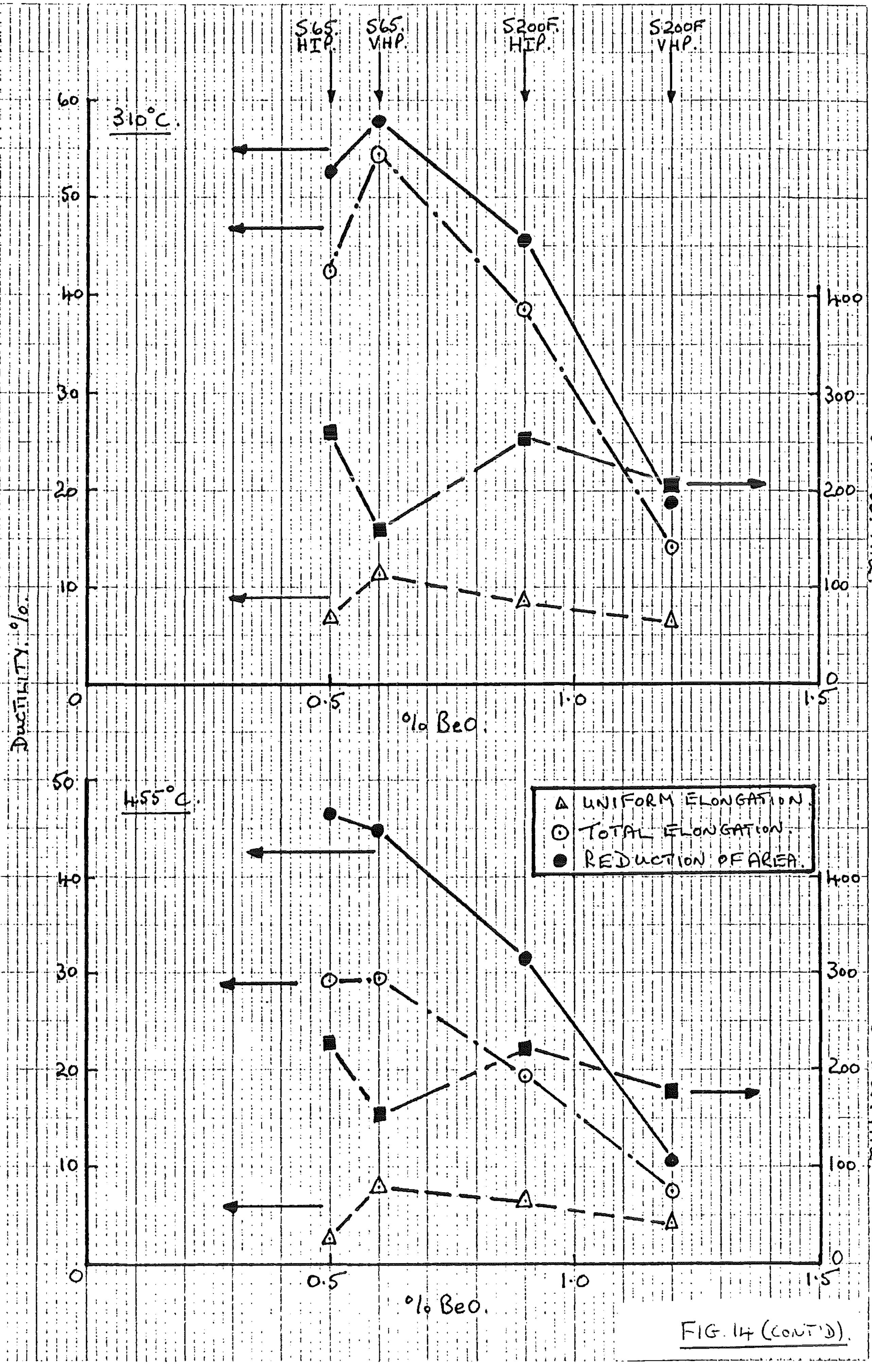

S65. HIP.
S65. VHP.
S200F. HIP.
S200F VHP.
310°C.
455°C.
DUCTILITY. %.
0.2% PROOF STRESS. MPa.
% BeO.
UNIFORM ELONGATION.
TOTAL ELONGATION.
REDUCTION OF AREA.
0
10
20
30
40
50
60
0.5
1.0
1.5
100
200
300
400

FIG. 14 (CONT'D).

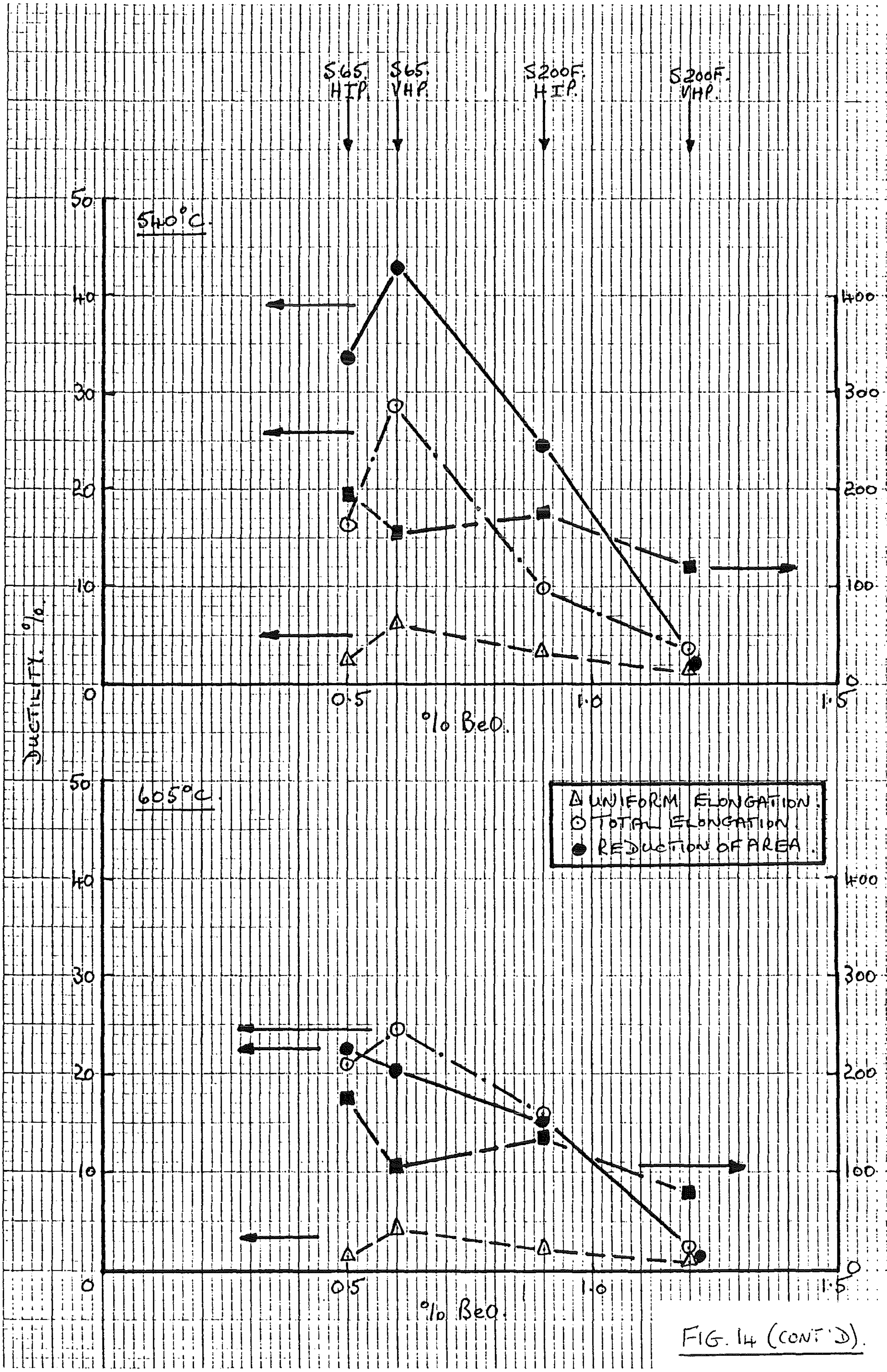

S65. HIP.
S65. VHP.
S200F. HIP.
S200F. VHP.
540°C.
605°C.
DUCTILITY. %
% BeO.
0.2% PROOF STRESS. MPa.
Δ UNIFORM ELONGATION.
⊙ TOTAL ELONGATION.
● REDUCTION OF AREA.

FIG. 14 (CONT'D).

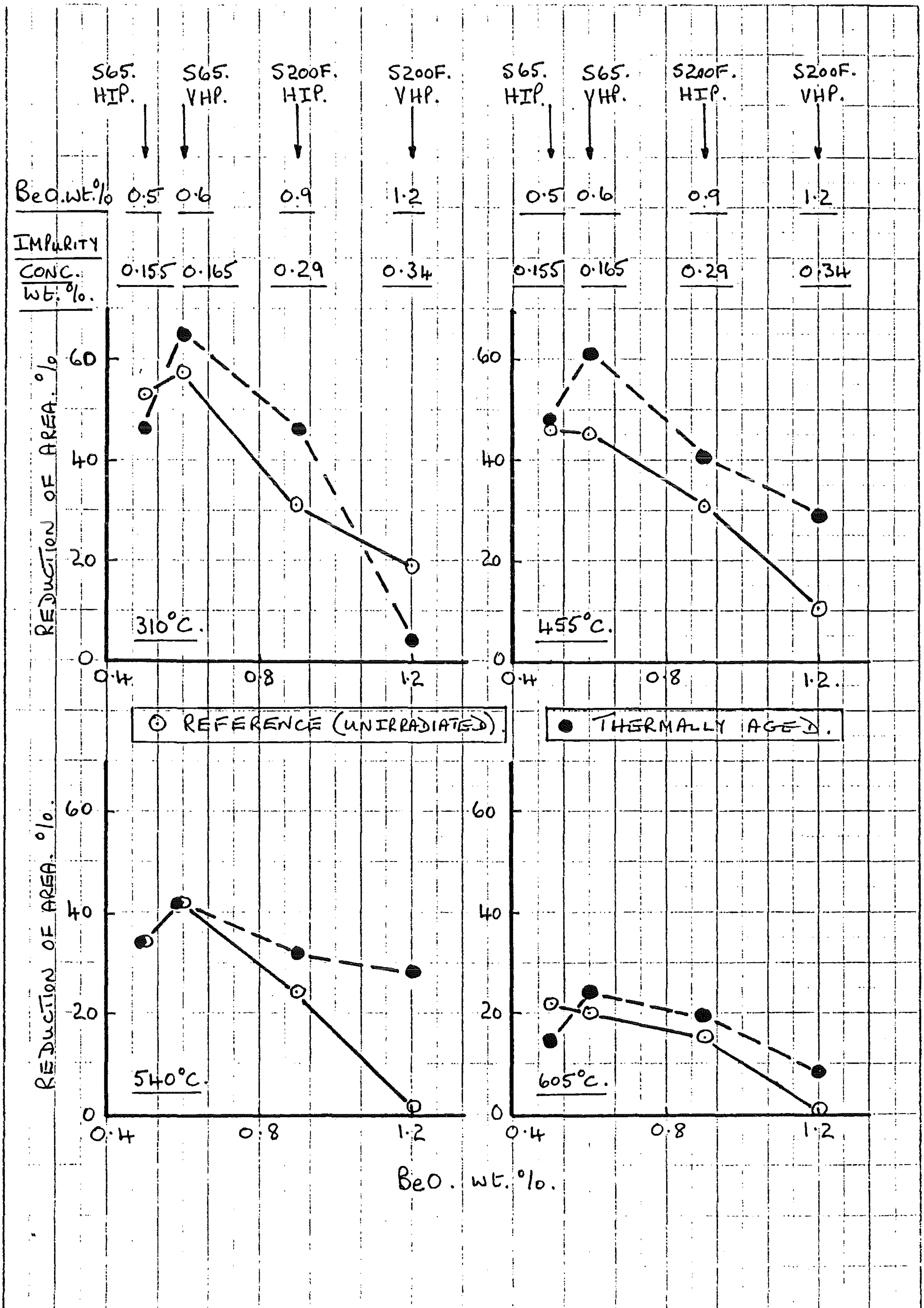
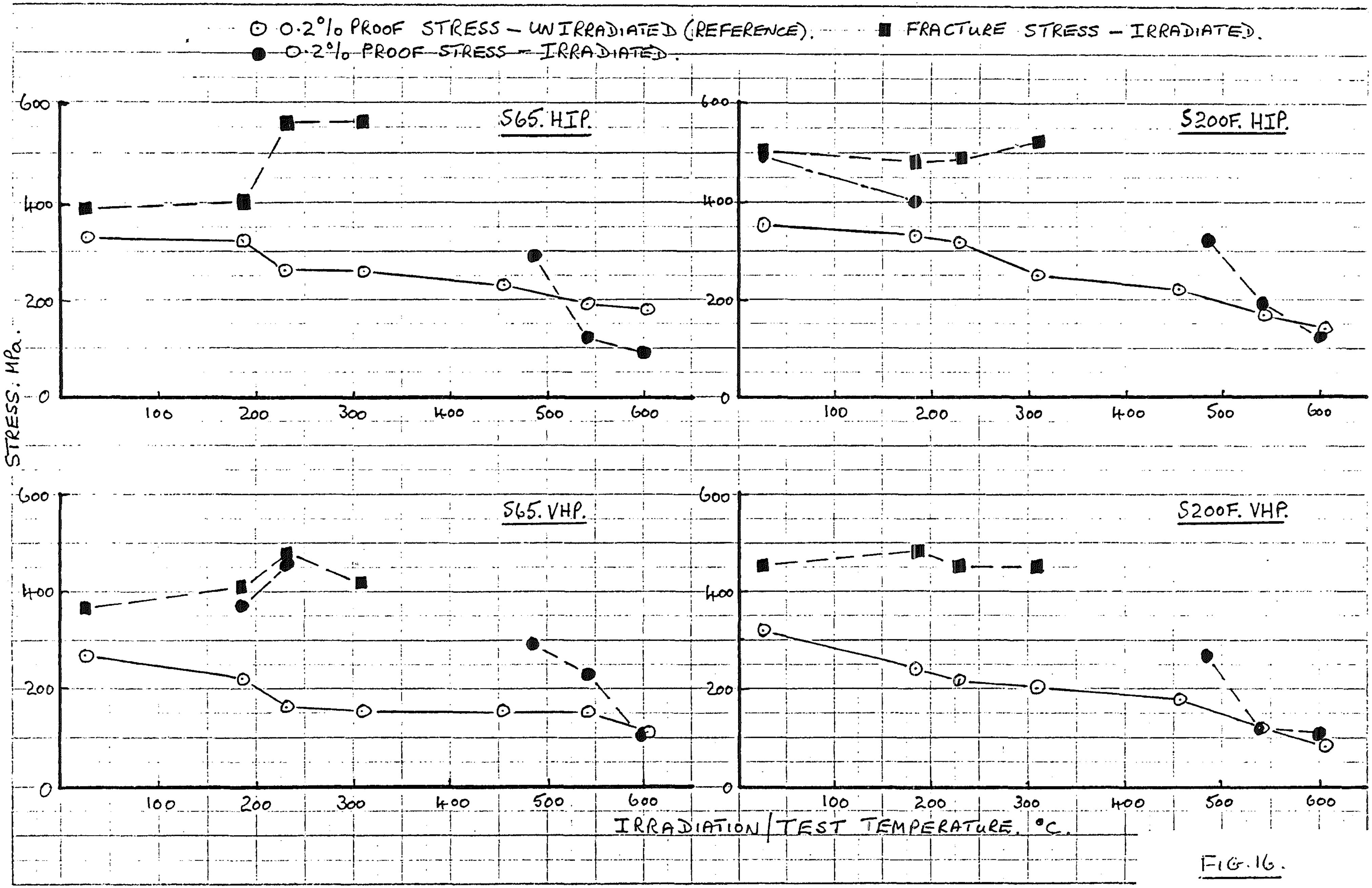
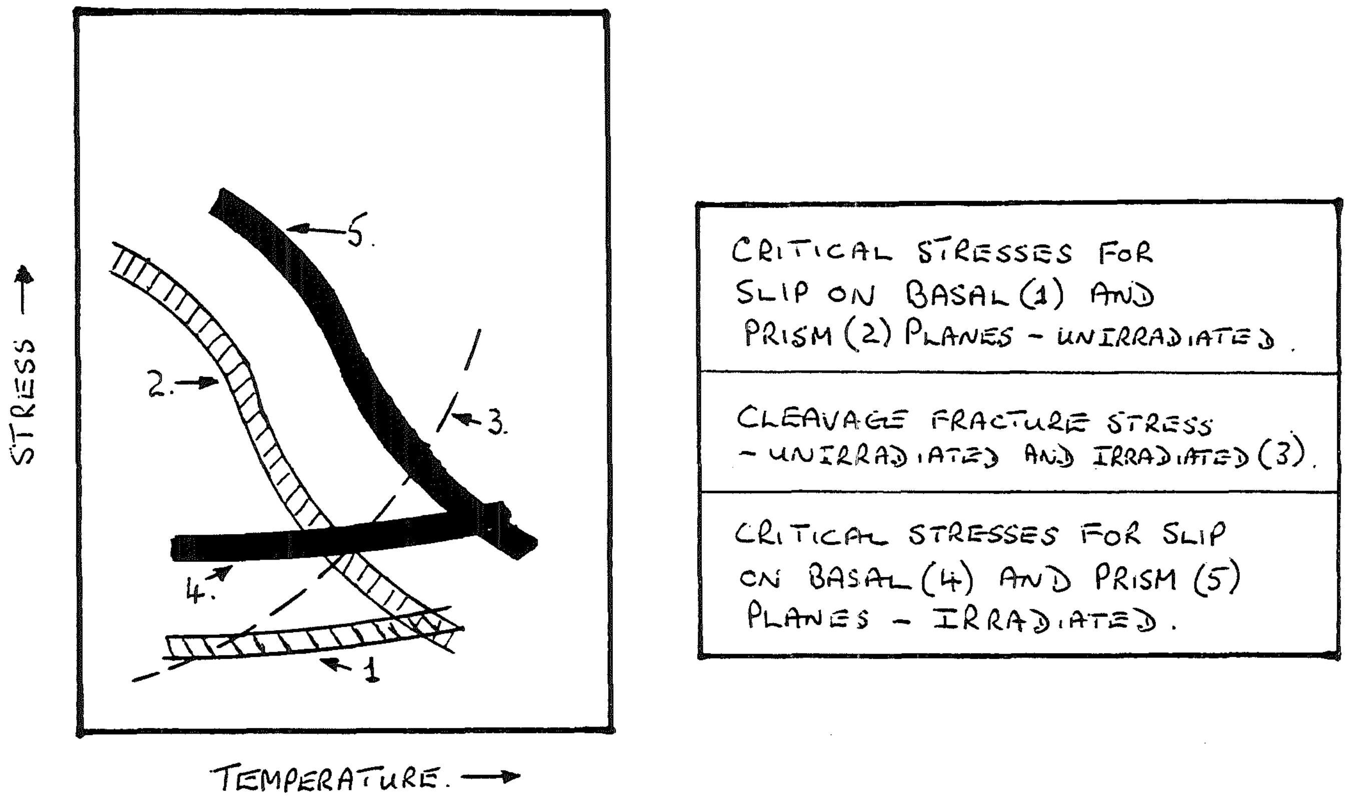

S65. HIP.
S65. VHP.
S200F. HIP.
S200F. VHP.
S65. HIP.
S65. VHP.
S200F. HIP.
S200F. VHP.
BeO.wt%
0·5
0·6
0·9
1·2
0·5
0·6
0·9
1·2
IMPURITY CONC. wt. %.
0·155
0·165
0·29
0·34
0·155
0·165
0·29
0·34
REDUCTION OF AREA. %
60
40
20
0
0·4
0·8
1·2
310°C.
455°C.
REFERENCE (UNIRRADIATED).
THERMALLY AGED.
540°C.
605°C.
BeO. wt. %.

FIG. 15.



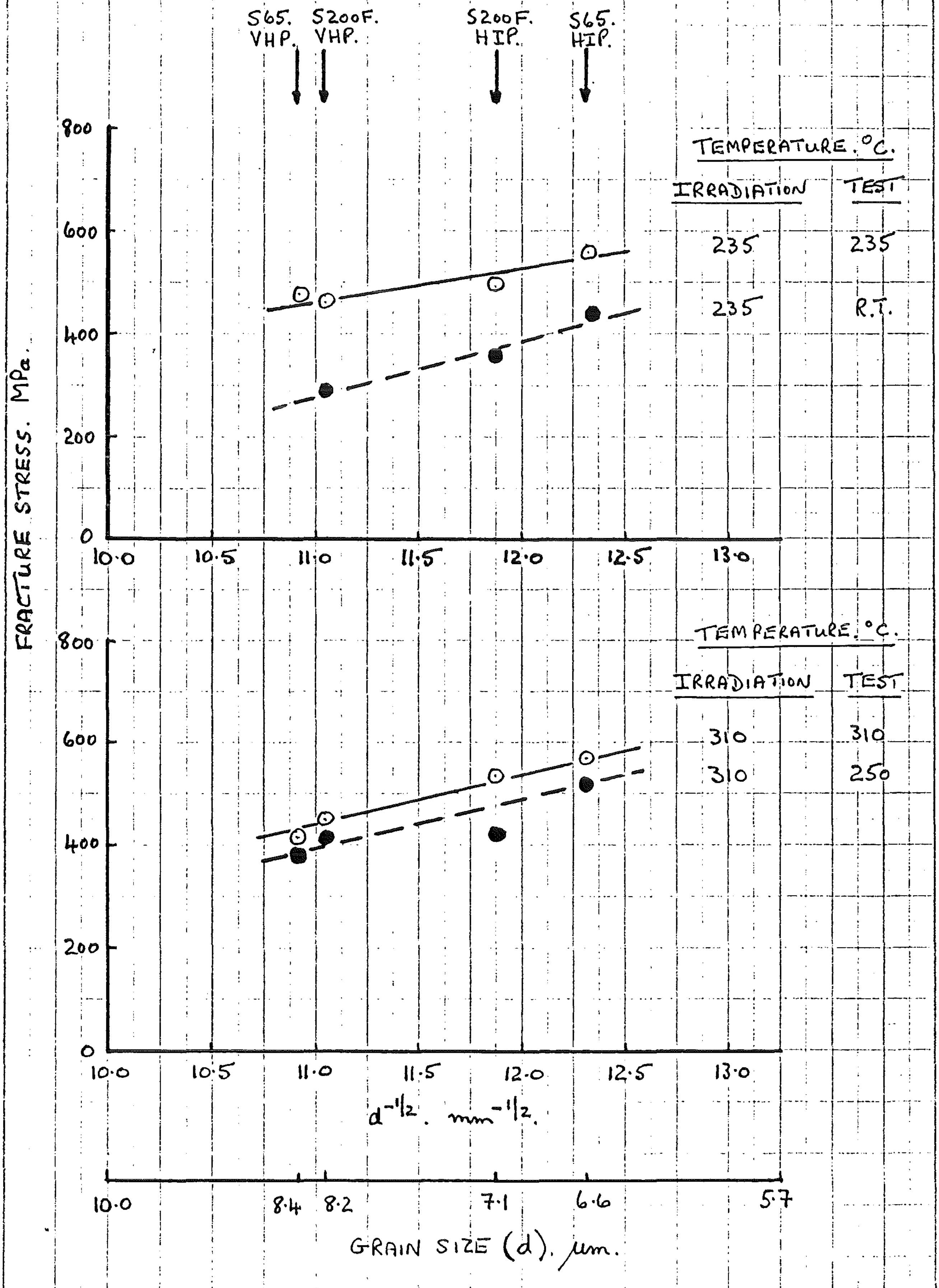
⊙ 0·2% PROOF STRESS – UNIRRADIATED (REFERENCE).
■ FRACTURE STRESS – IRRADIATED.
● 0·2% PROOF STRESS – IRRADIATED.
S65. HIP.
S200F. HIP.
S65. VHP.
S200F. VHP.
STRESS: MPa.
IRRADIATION/TEST TEMPERATURE. °C.

FIG. 16.



5.
2.
3.
4.
1
STRESS
TEMPERATURE.
CRITICAL STRESSES FOR SLIP ON BASAL (1) AND PRISM (2) PLANES - UNIRRADIATED.
CLEAVAGE FRACTURE STRESS - UNIRRADIATED AND IRRADIATED (3).
CRITICAL STRESSES FOR SLIP ON BASAL (4) AND PRISM (5) PLANES - IRRADIATED.

FIG. 17.



S65. VHP.
S200F. VHP.
S200F. HIP.
S65. HIP.
TEMPERATURE. °C.
IRRADIATION
TEST
235
235
235
R.T.
800
600
400
200
0
10·0
10·5
11·0
11·5
12·0
12·5
13·0
FRACTURE STRESS. MPa.
TEMPERATURE. °C.
IRRADIATION
TEST
310
310
310
250
d^-1/2. mm^-1/2.
10.0
8·4
8·2
7·1
6·6
5·7
GRAIN SIZE (d). µm.

FIG. 18

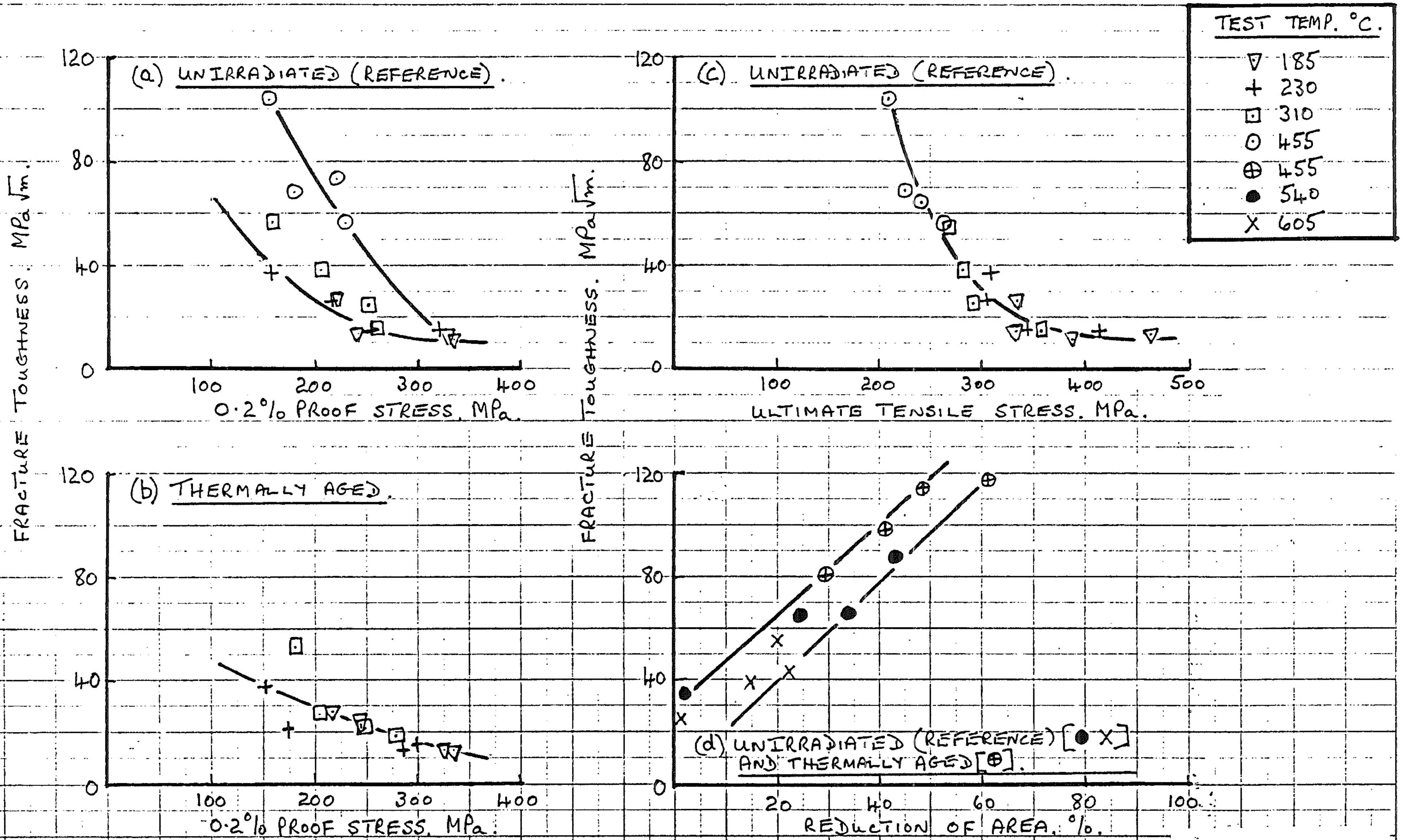

TEST TEMP. °C.
185
230
310
455
455
540
605
(a) UNIRRADIATED (REFERENCE).
0·2% PROOF STRESS. MPa.
(b) THERMALLY AGED.
0·2% PROOF STRESS. MPa.
(c) UNIRRADIATED (REFERENCE).
ULTIMATE TENSILE STRESS. MPa.
(d) UNIRRADIATED (REFERENCE) [● X] AND THERMALLY AGED [⊕].
REDUCTION OF AREA. %.
FRACTURE TOUGHNESS. MPa√m.
FRACTURE TOUGHNESS. MPa√m.
0
40
80
120
100
200
300
400
500
20
40
60
80
100

FIG. 19.

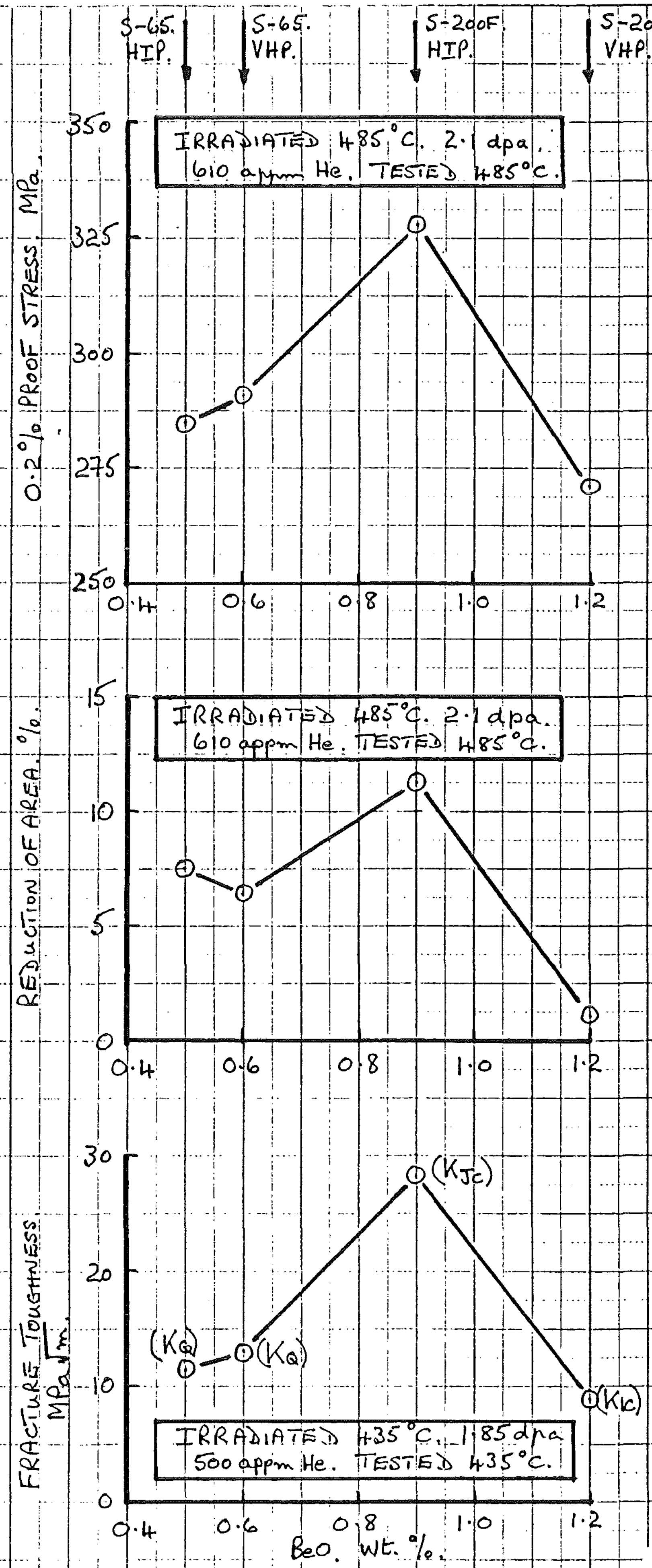
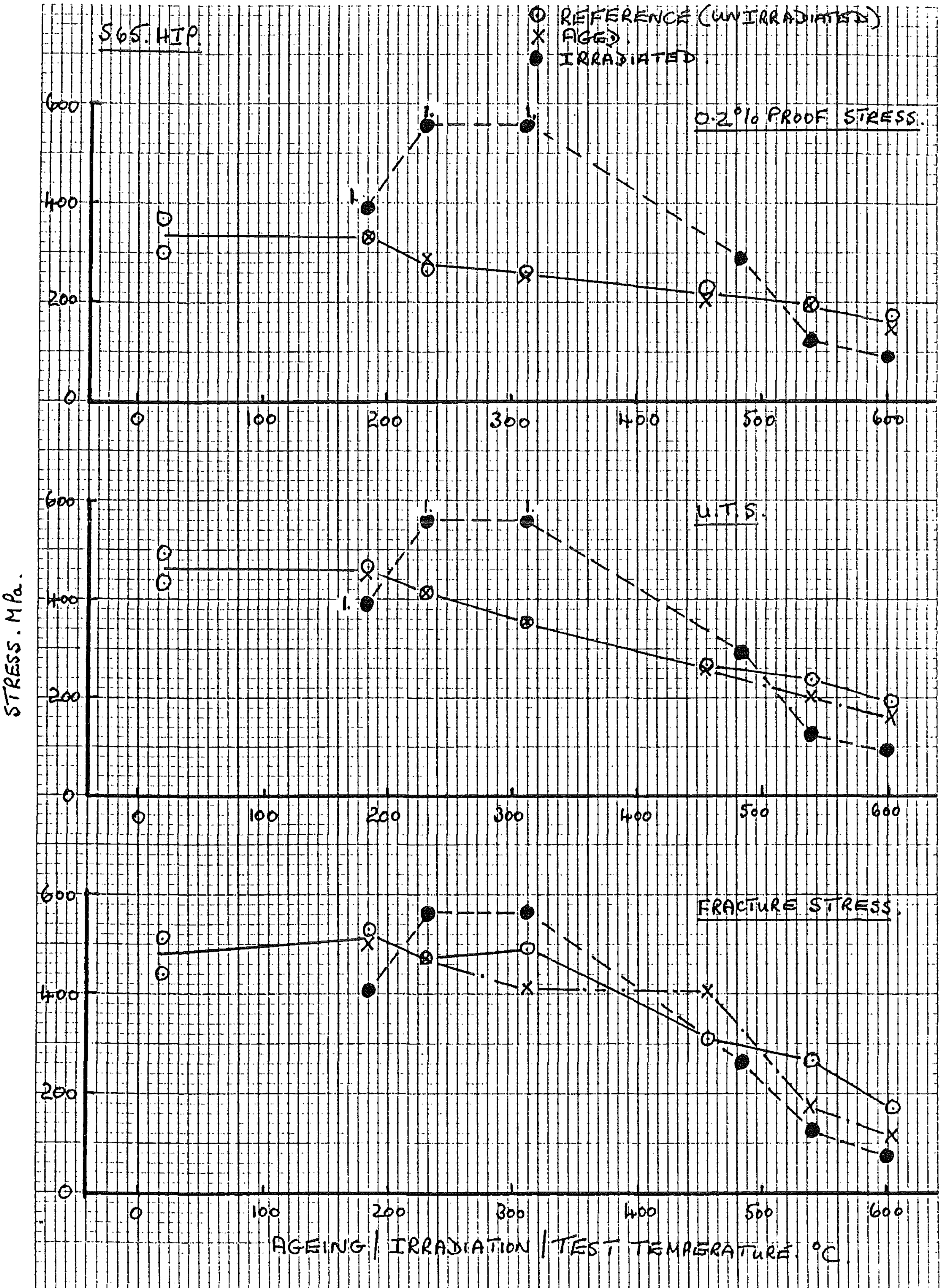

S-65. HIP.
S-65. VHP.
S-200F. HIP.
S-200F. VHP.
IRRADIATED 485°C. 2·1 dpa. 610 appm He. TESTED 485°C.
0·2% PROOF STRESS. MPa.
350
325
300
275
250
0·4
0·6
0·8
1·0
1·2
IRRADIATED 485°C. 2·1 dpa. 610 appm He. TESTED 485°C.
REDUCTION OF AREA. %.
15
10
5
0
(KJc)
(KQ)
(KQ)
(KIc)
FRACTURE TOUGHNESS. MPa√m.
30
20
10
0
IRRADIATED 435°C. 1·85 dpa 500 appm He. TESTED 435°C.
BeO. wt. %.

FIG. 20

Appendix A

Effects of Ageing, Irradiation and Test Temperature on the Tensile Properties of S-65. HIP (Fig. A1), S-65. VHP (Fig. A2), S-200F. HIP (Fig. A3) and S-200F. VHP (Fig. A4) Beryllium.



. FRACTURE WITHIN THE LINEAR REGION (ELASTIC RANGE).

FIG. A1.

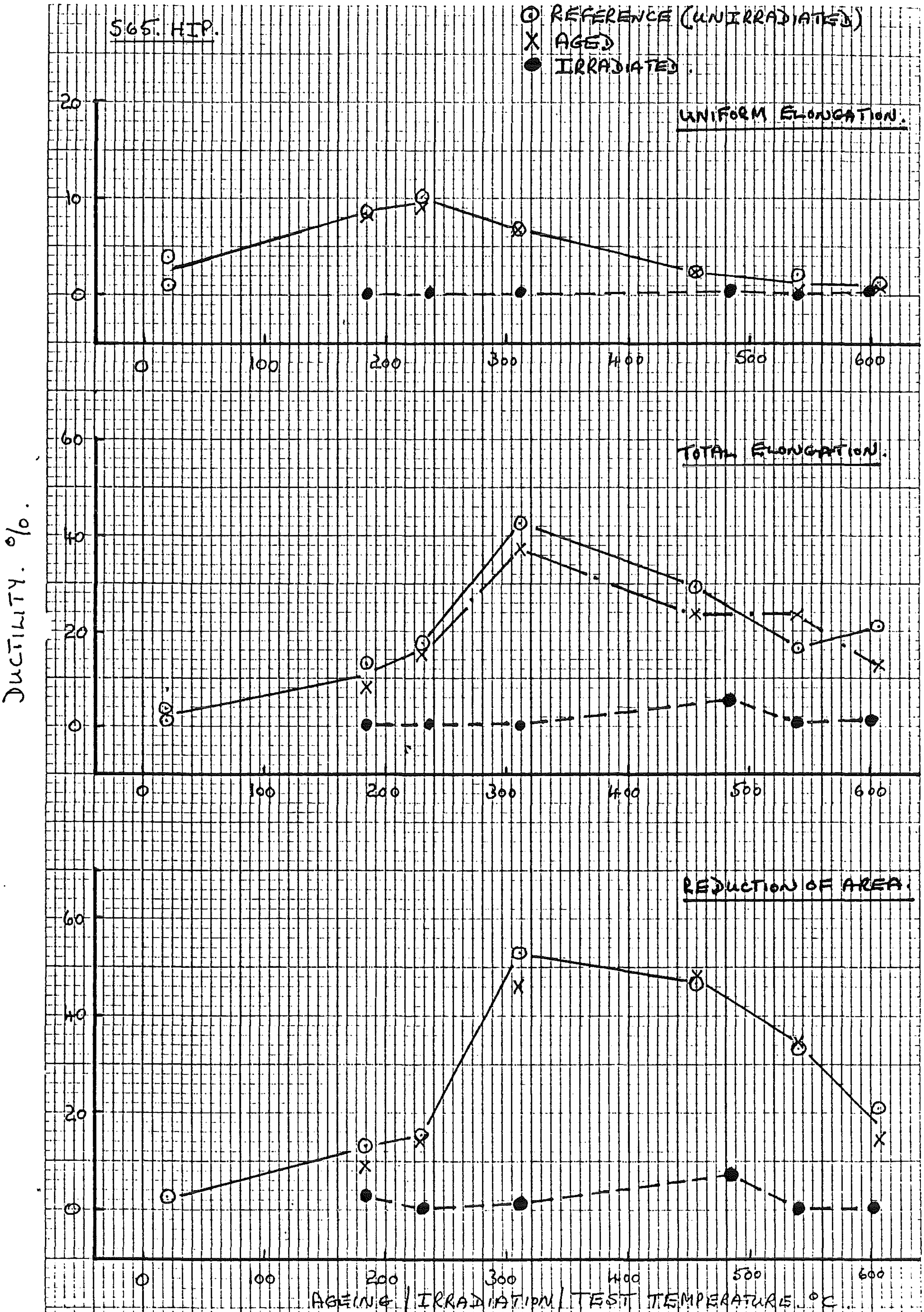
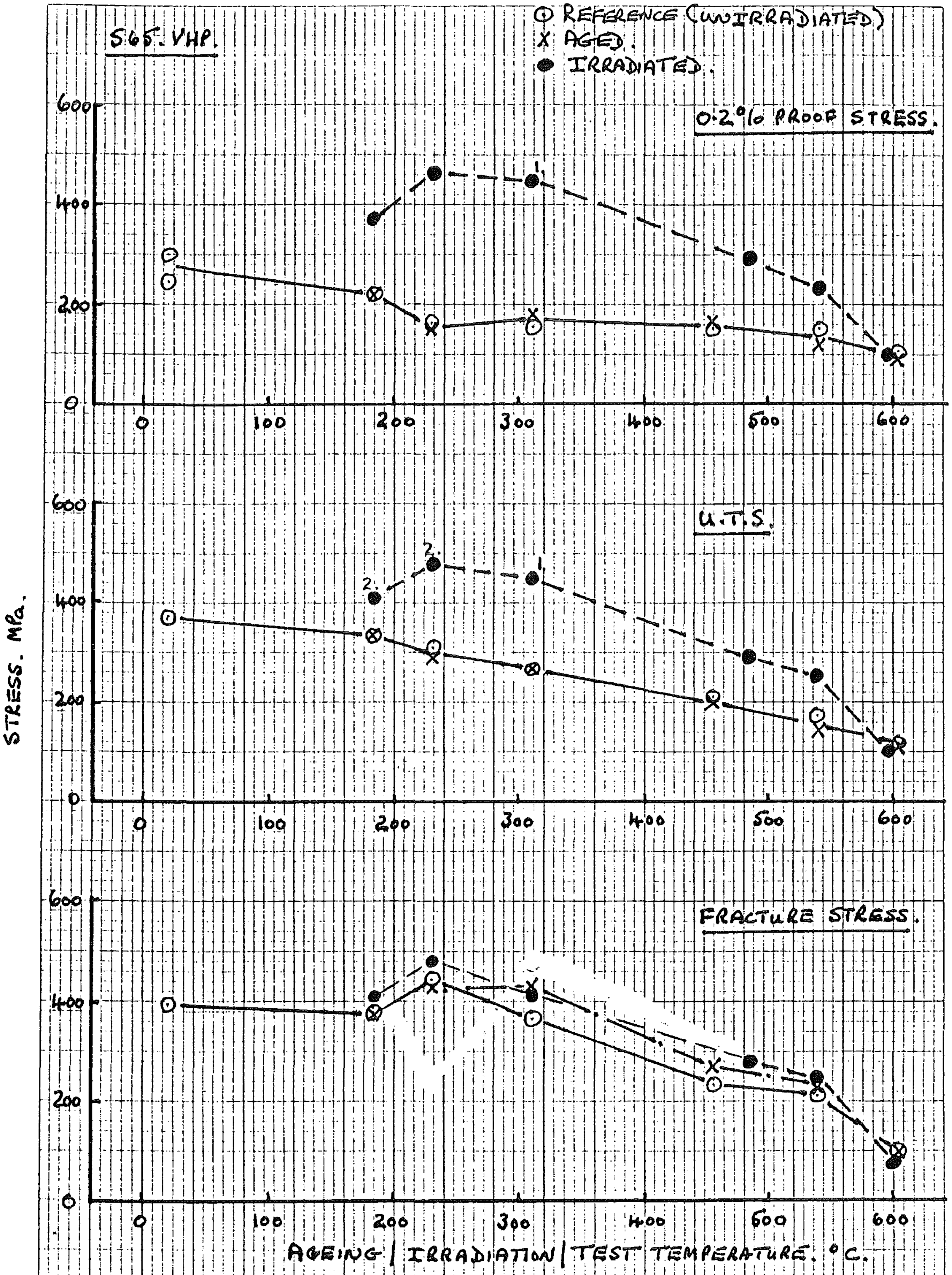

565. HIP.
⊙ REFERENCE (UNIRRADIATED)
X AGED
● IRRADIATED.
UNIFORM ELONGATION.
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY. %.
0
10
20
40
60
100
200
300
400
500
600
AGEING / IRRADIATION / TEST TEMPERATURE °C

FIG. A1 (CONTD.)



1. FRACTURE WITHIN THE LINEAR REGION (ELASTIC RANGE).
2. FRACTURE AT MAXIMUM LOAD (ZERO NON-UNIFORM DEFORMATION).

FIG. A2.

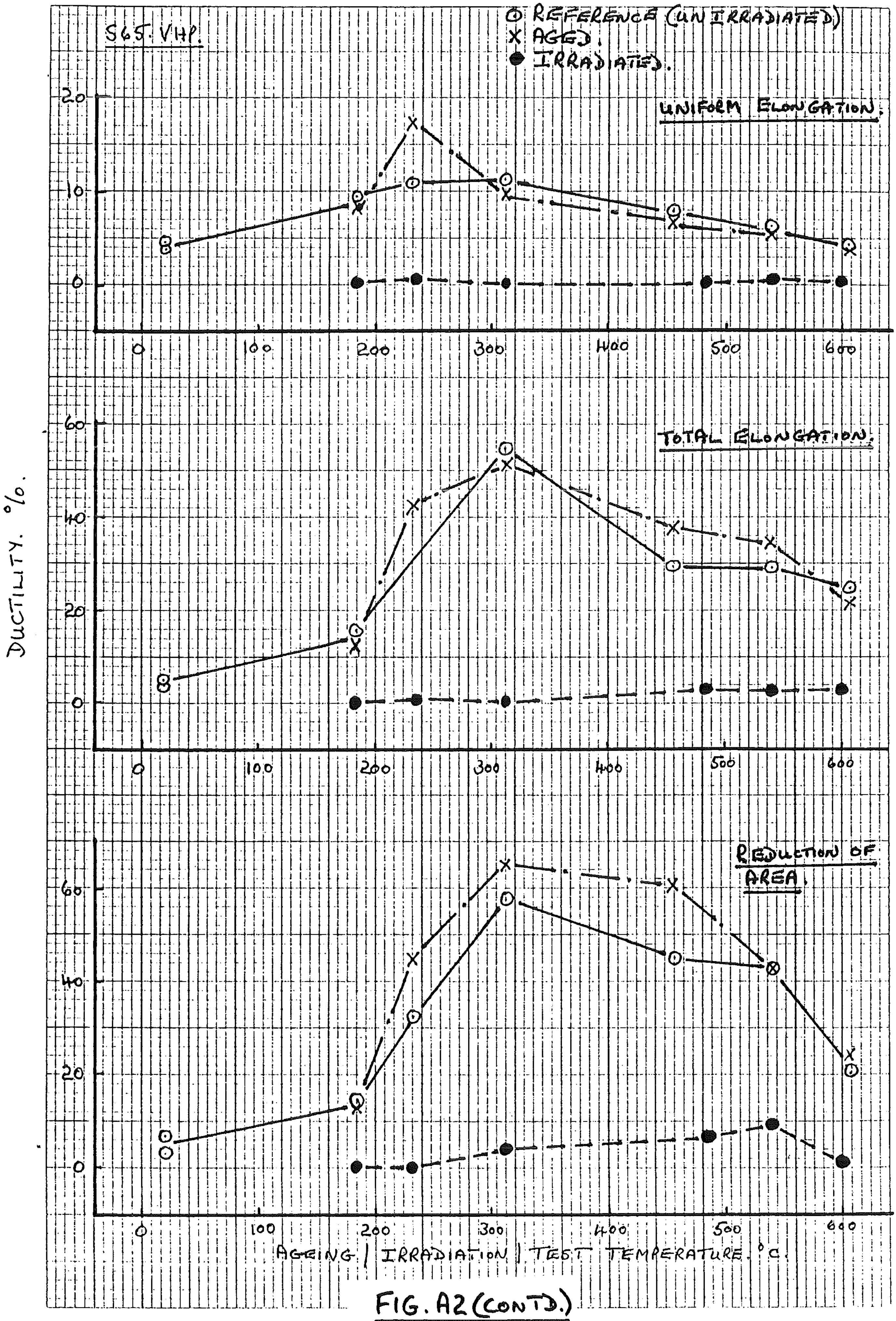
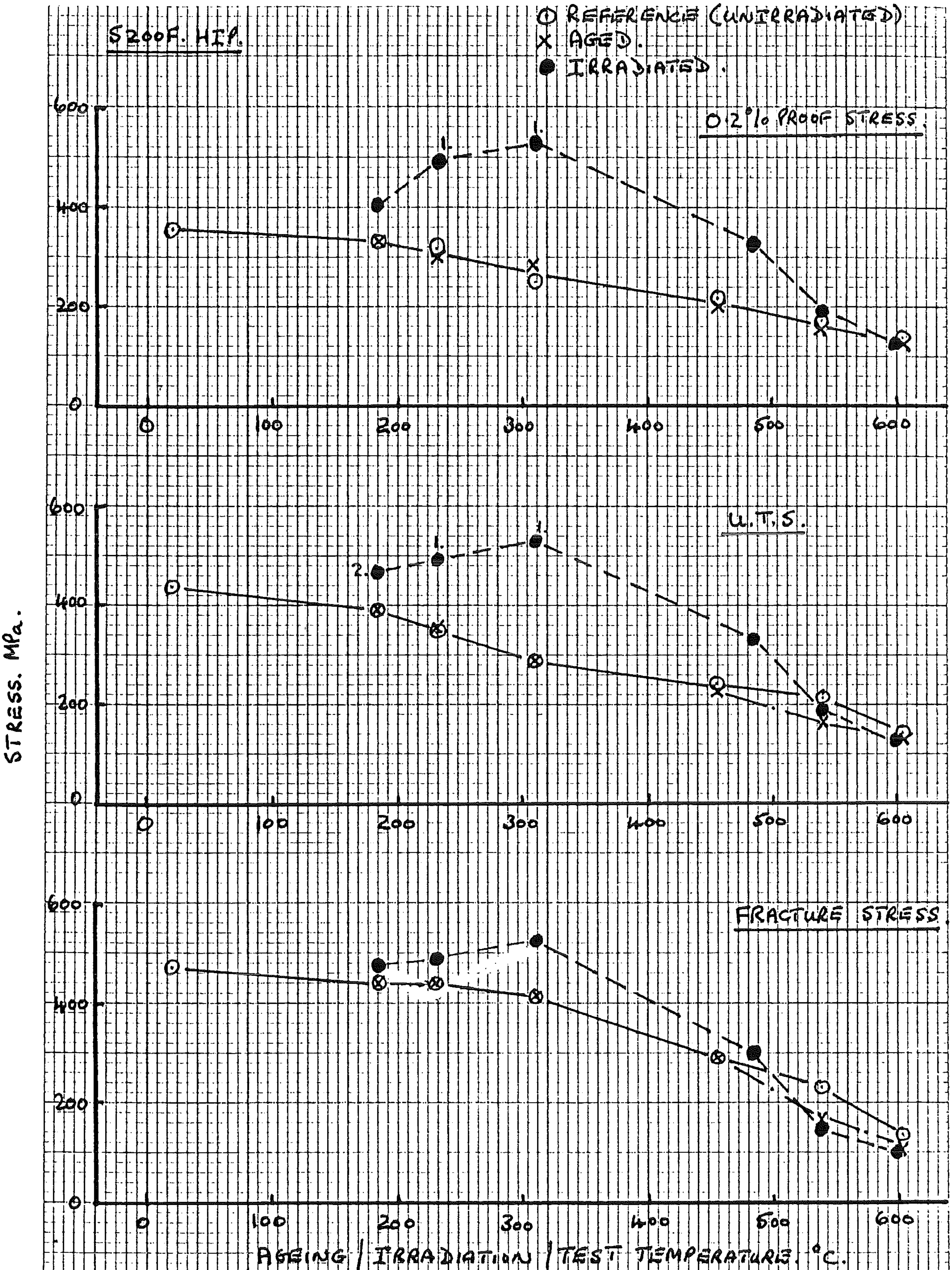

S65 VHP.
⊙ REFERENCE (UNIRRADIATED)
X AGED.
● IRRADIATED.
UNIFORM ELONGATION.
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY. %.
0
10
20
40
60
0
100
200
300
400
500
600
AGEING / IRRADIATION / TEST TEMPERATURE °C.

FIG. A2 (CONTD.)



1. FRACTURE WITHIN THE LINEAR REGION (ELASTIC RANGE).
2. FRACTURE AT MAXIMUM LOAD (ZERO NON-UNIFORM DEFORMATION).

FIG. A3.

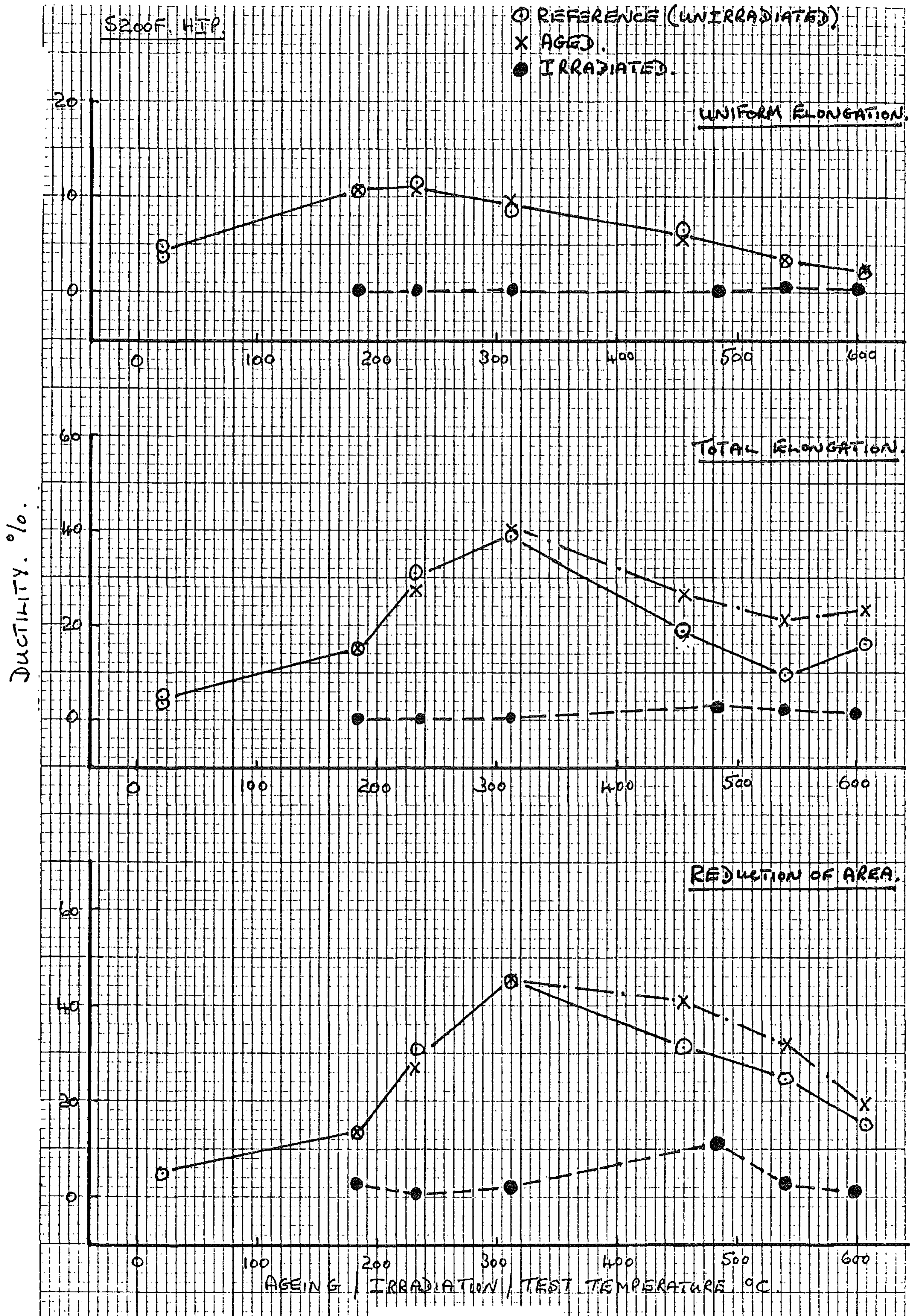

S200F. HIP.
REFERENCE (UNIRRADIATED)
X AGED.
IRRADIATED.
UNIFORM ELONGATION.
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY. %.
AGEING / IRRADIATION / TEST TEMPERATURE °C.
0
10
20
40
60
100
200
300
400
500
600

FIG. A3 (CONTD).

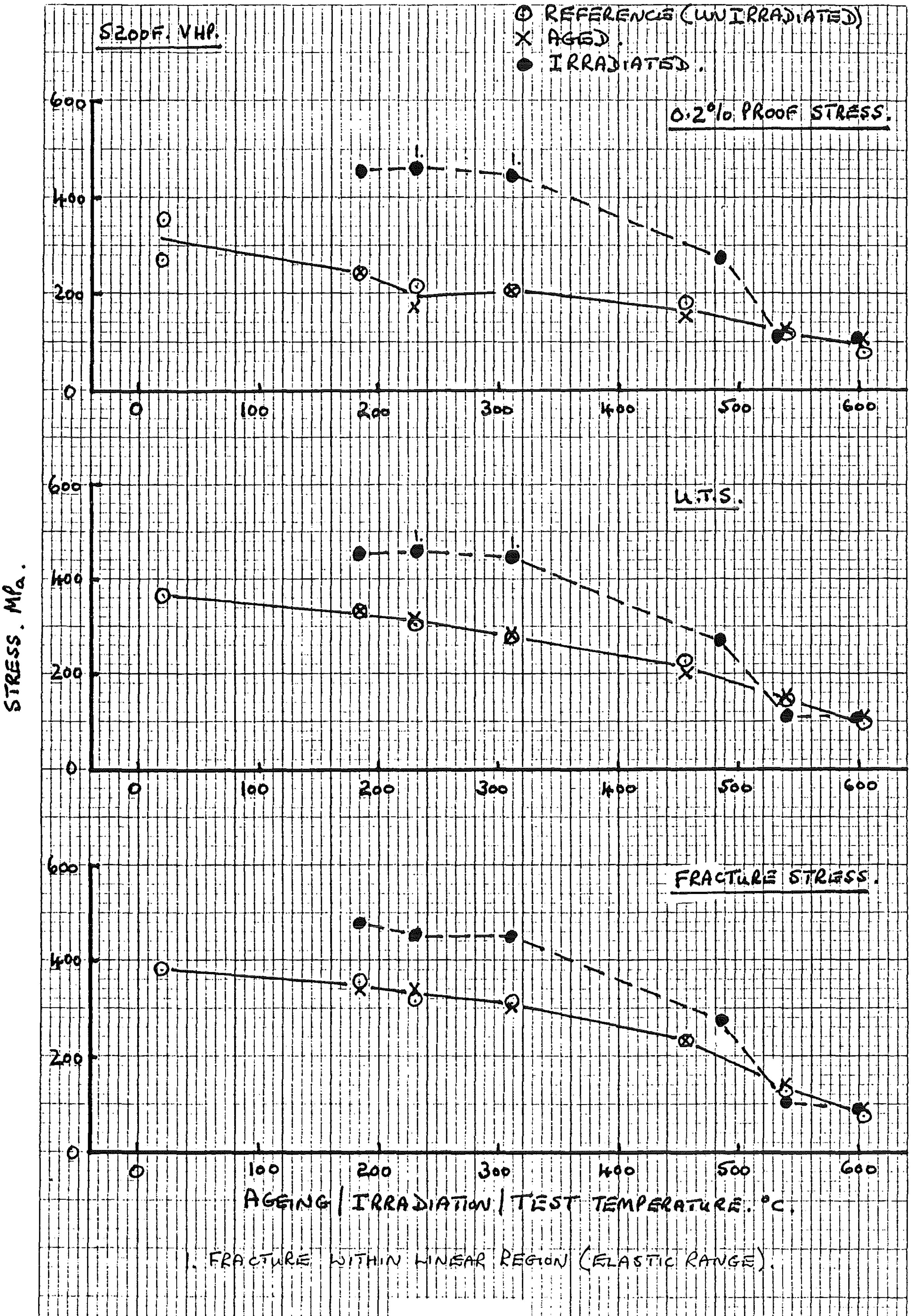

S200F. VHP.
⊙ REFERENCE (UNIRRADIATED)
X AGED.
● IRRADIATED.
0.2% PROOF STRESS.
U.T.S.
FRACTURE STRESS.
STRESS. MPa.
600
400
200
0
100
200
300
400
500
600
1.
AGEING | IRRADIATION | TEST TEMPERATURE. °C.
1. FRACTURE WITHIN LINEAR REGION (ELASTIC RANGE).

FIG. A4

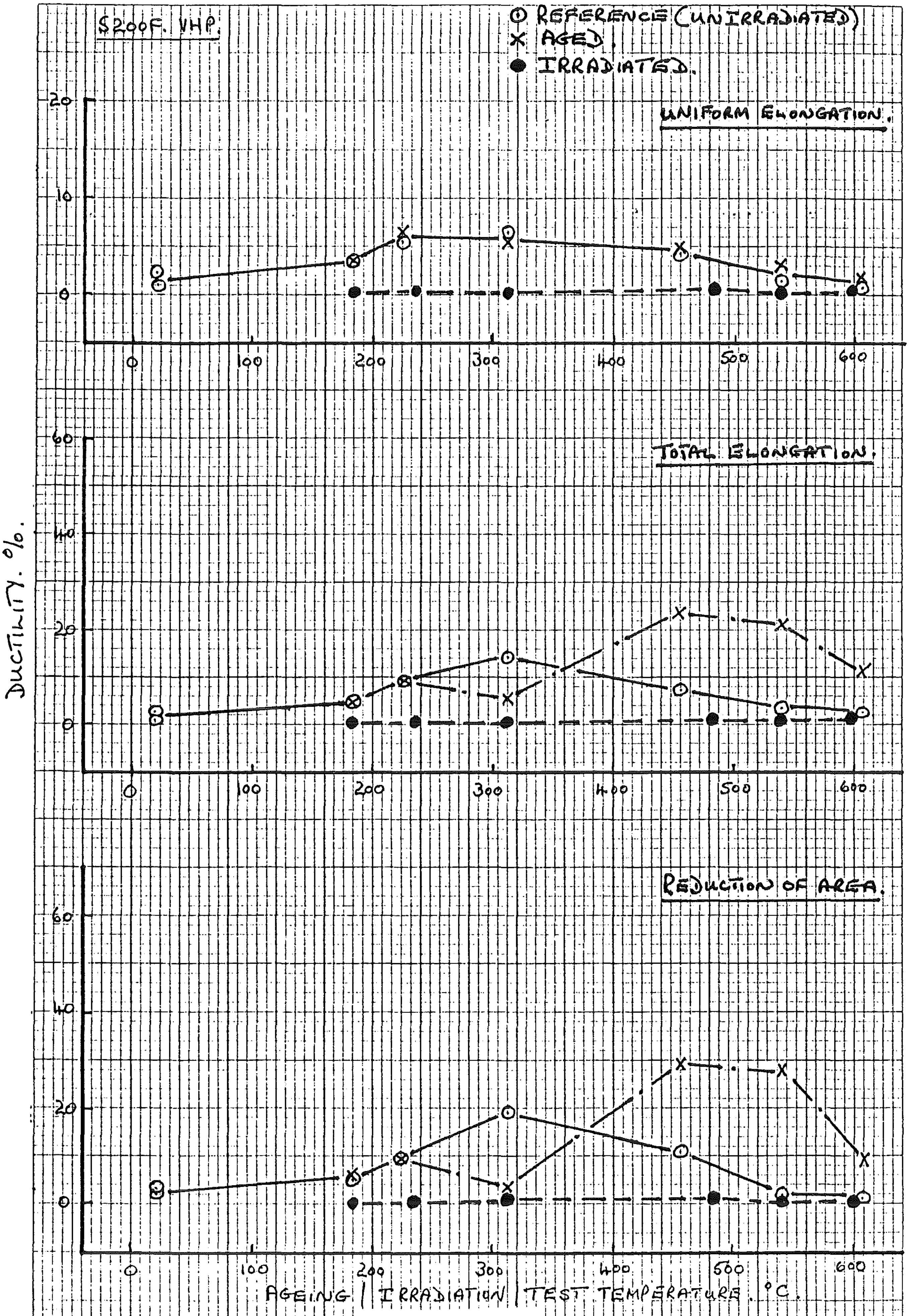
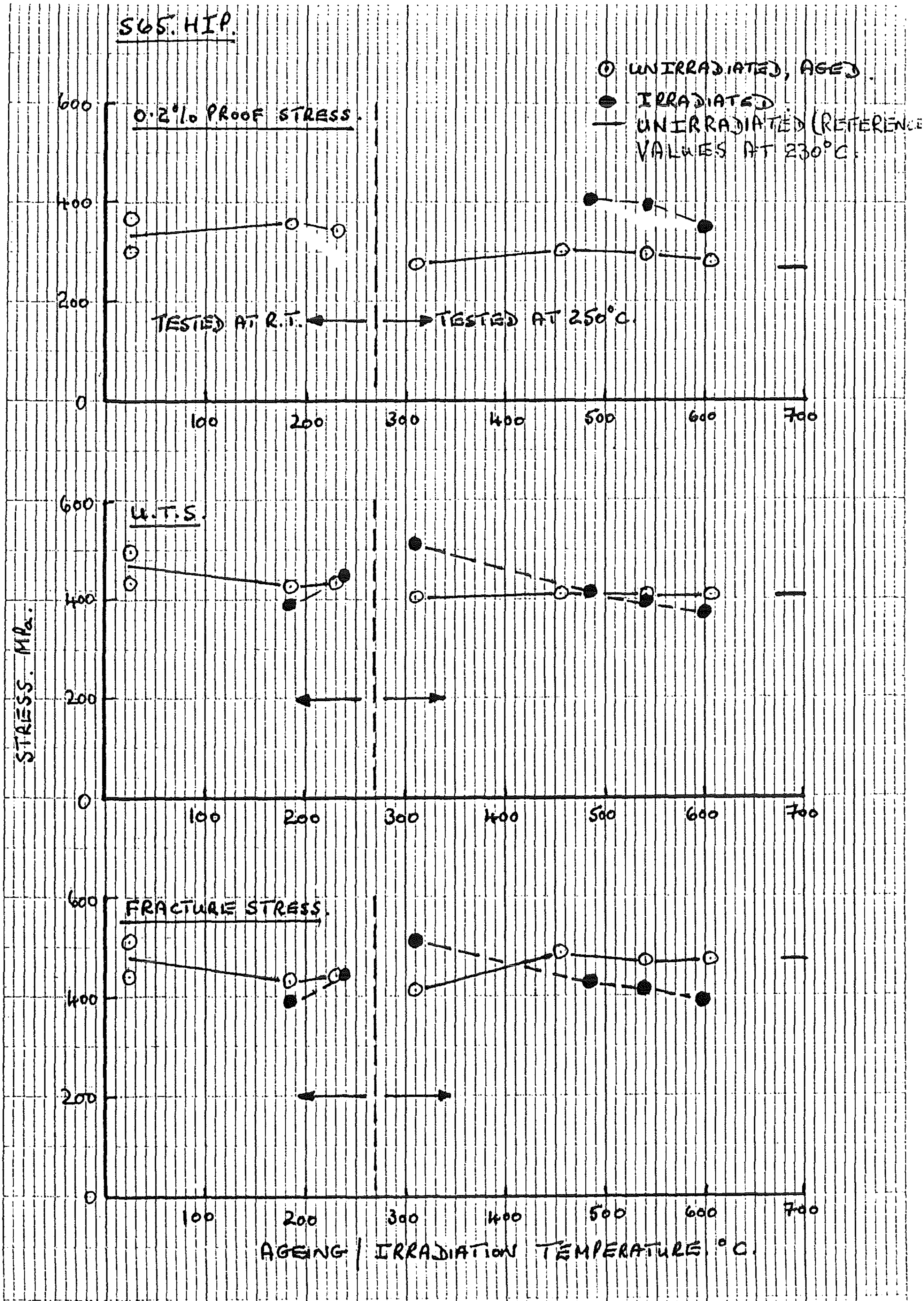

S200F. VHP.
⊙ REFERENCE (UNIRRADIATED)
X AGED.
● IRRADIATED.
UNIFORM ELONGATION.
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY. %.
0
10
20
40
60
100
200
300
400
500
600
AGEING / IRRADIATION / TEST TEMPERATURE. °C.

FIG. A4 (CONTD)

Appendix B

Effects of Ageing and Irradiation at 185 and 230/235 C on the Ambient Temperature Tensile Properties and at 310, 455/485, 540 and 605 C on the Tensile Properties at 250 C of the S-65, HIP (Fig. B1), S-65, VHP (Fig. B2), S-200F, HIP (Fig. B3) and S-200F, VHP (Fig. B4) Beryllium.



S65. HIP.
UNIRRADIATED, AGED.
IRRADIATED.
UNIRRADIATED (REFERENCE VALUES AT 230°C.
0.2% PROOF STRESS.
TESTED AT R.T.
TESTED AT 250°C.
U.T.S.
FRACTURE STRESS.
STRESS. MPa.
AGEING / IRRADIATION TEMPERATURE °C.
0
200
400
600
100
300
500
700

FIG. B1.

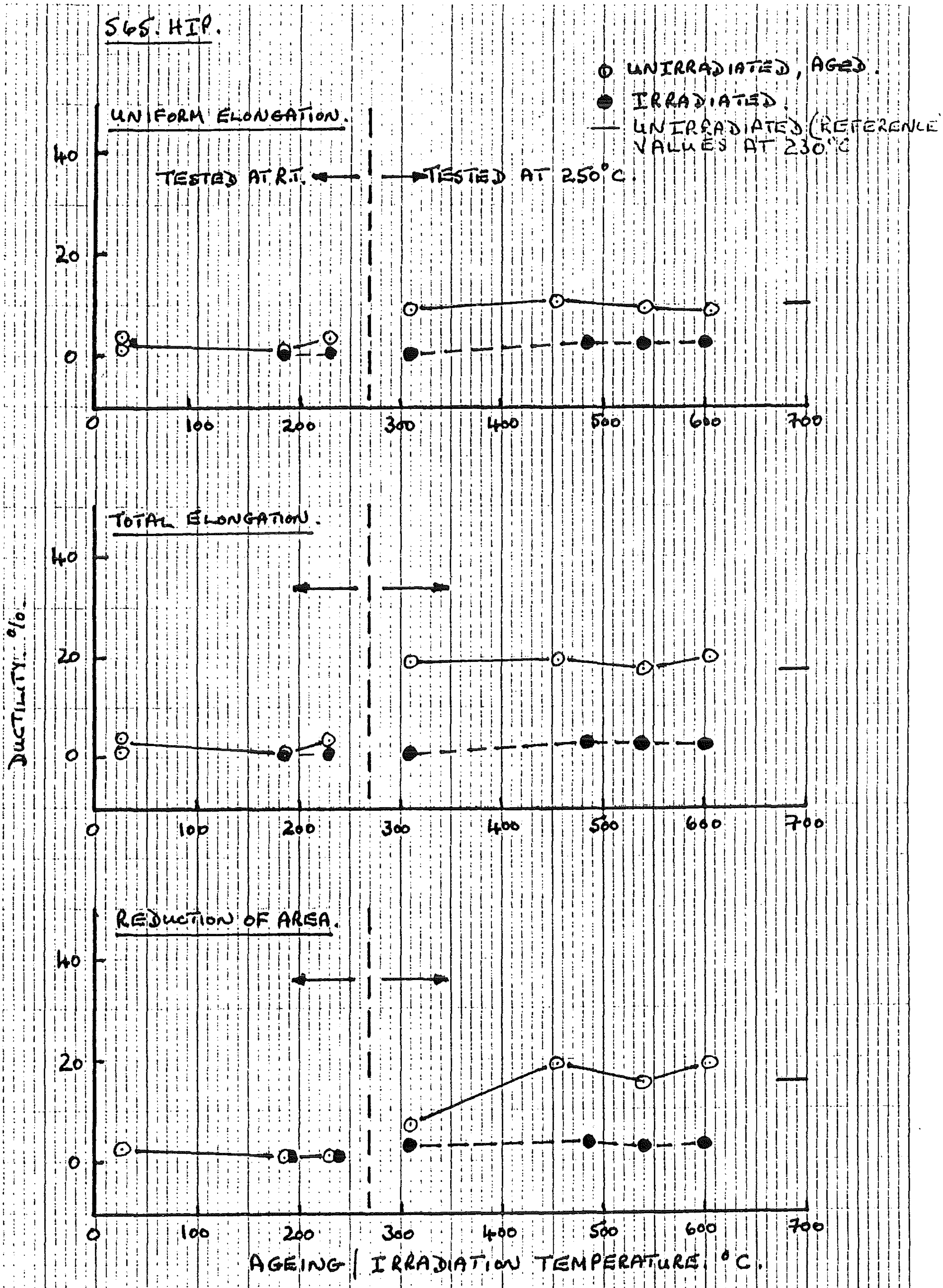

565. HIP.
UNIRRADIATED, AGED.
IRRADIATED.
UNIRRADIATED (REFERENCE VALUES AT 230°C
UNIFORM ELONGATION.
TESTED AT R.T.
TESTED AT 250°C.
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY: %.
0
20
40
100
200
300
400
500
600
700
AGEING / IRRADIATION TEMPERATURE °C.

FIG. B1. (CONTD.)

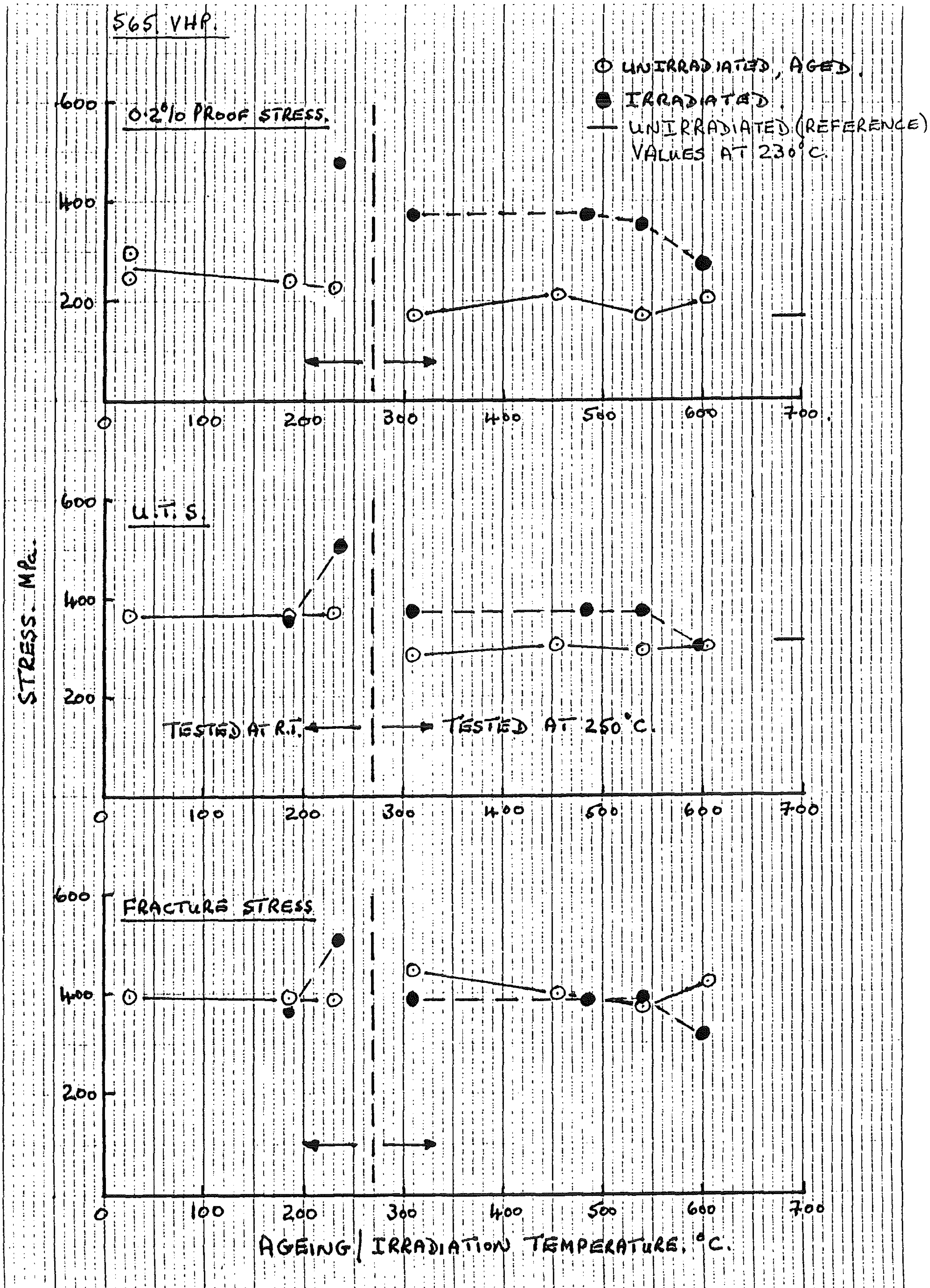

565 VHP.
UNIRRADIATED, AGED.
IRRADIATED.
UNIRRADIATED (REFERENCE) VALUES AT 230°C.
0·2% PROOF STRESS.
U.T.S.
FRACTURE STRESS
STRESS. MPa.
600
400
200
0
100
200
300
400
500
600
700
TESTED AT R.T.
TESTED AT 250°C.
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. B2.

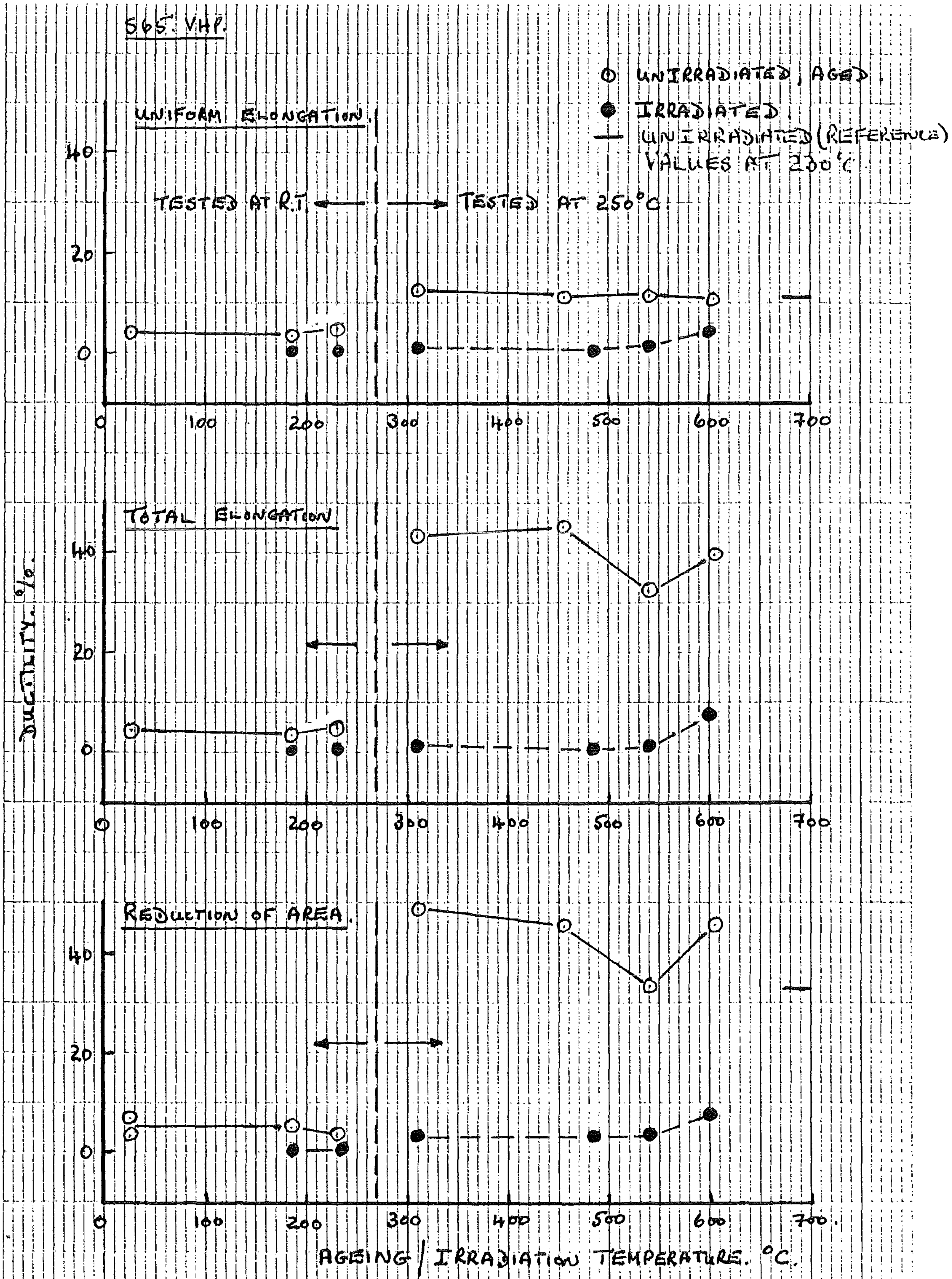

565. VHP.
UNIRRADIATED, AGED.
IRRADIATED.
UNIRRADIATED (REFERENCE) VALUES AT 250°C
UNIFORM ELONGATION.
TESTED AT R.T.
TESTED AT 250°C.
TOTAL ELONGATION
REDUCTION OF AREA.
DUCTILITY. %.
0
20
40
0
100
200
300
400
500
600
700
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. B2 (CONTD.)

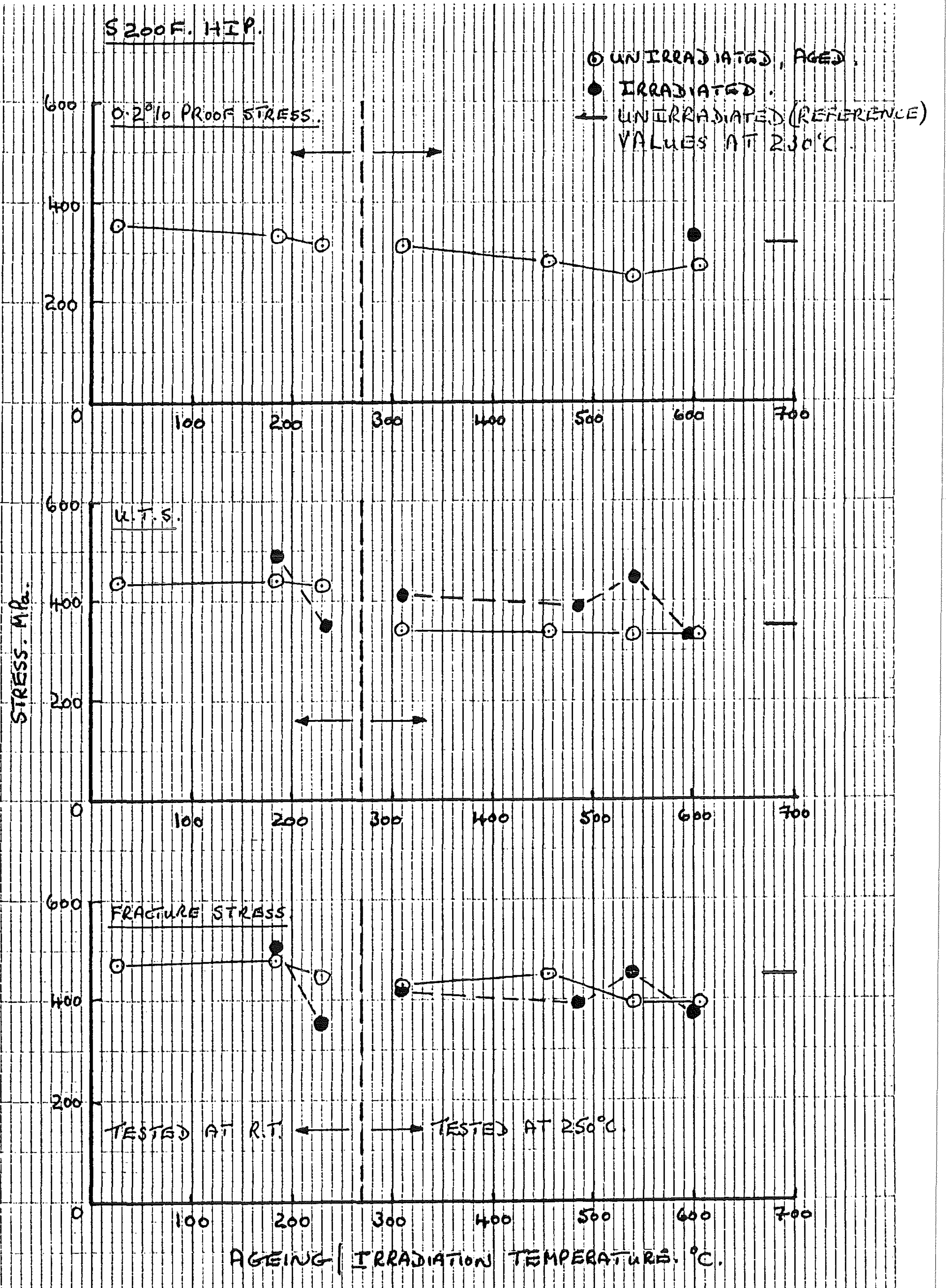

S200F. HIP.
⊙ UNIRRADIATED, AGED.
● IRRADIATED.
— UNIRRADIATED (REFERENCE) VALUES AT 230°C
0.2% PROOF STRESS.
U.T.S.
FRACTURE STRESS.
TESTED AT R.T.
TESTED AT 250°C.
STRESS. MPa.
AGEING | IRRADIATION TEMPERATURE. °C.
0
100
200
300
400
500
600
700

FIG. B3

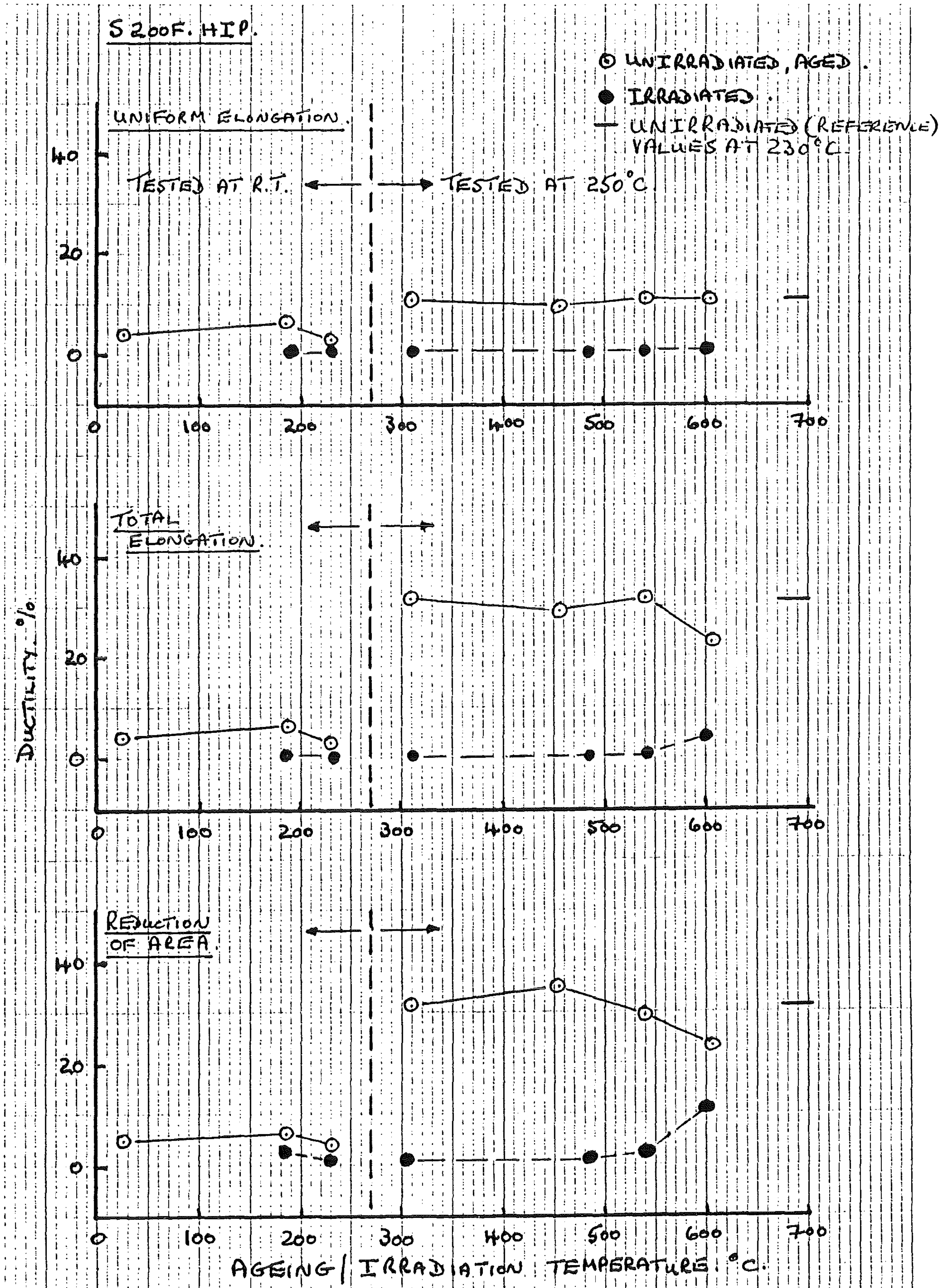

S 200F. HIP.
⊙ UNIRRADIATED, AGED.
● IRRADIATED.
— UNIRRADIATED (REFERENCE) VALUES AT 230°C.
UNIFORM ELONGATION.
TESTED AT R.T.
TESTED AT 250°C
TOTAL ELONGATION.
REDUCTION OF AREA.
DUCTILITY. %
0
20
40
0
100
200
300
400
500
600
700
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. B3 (CONTD).

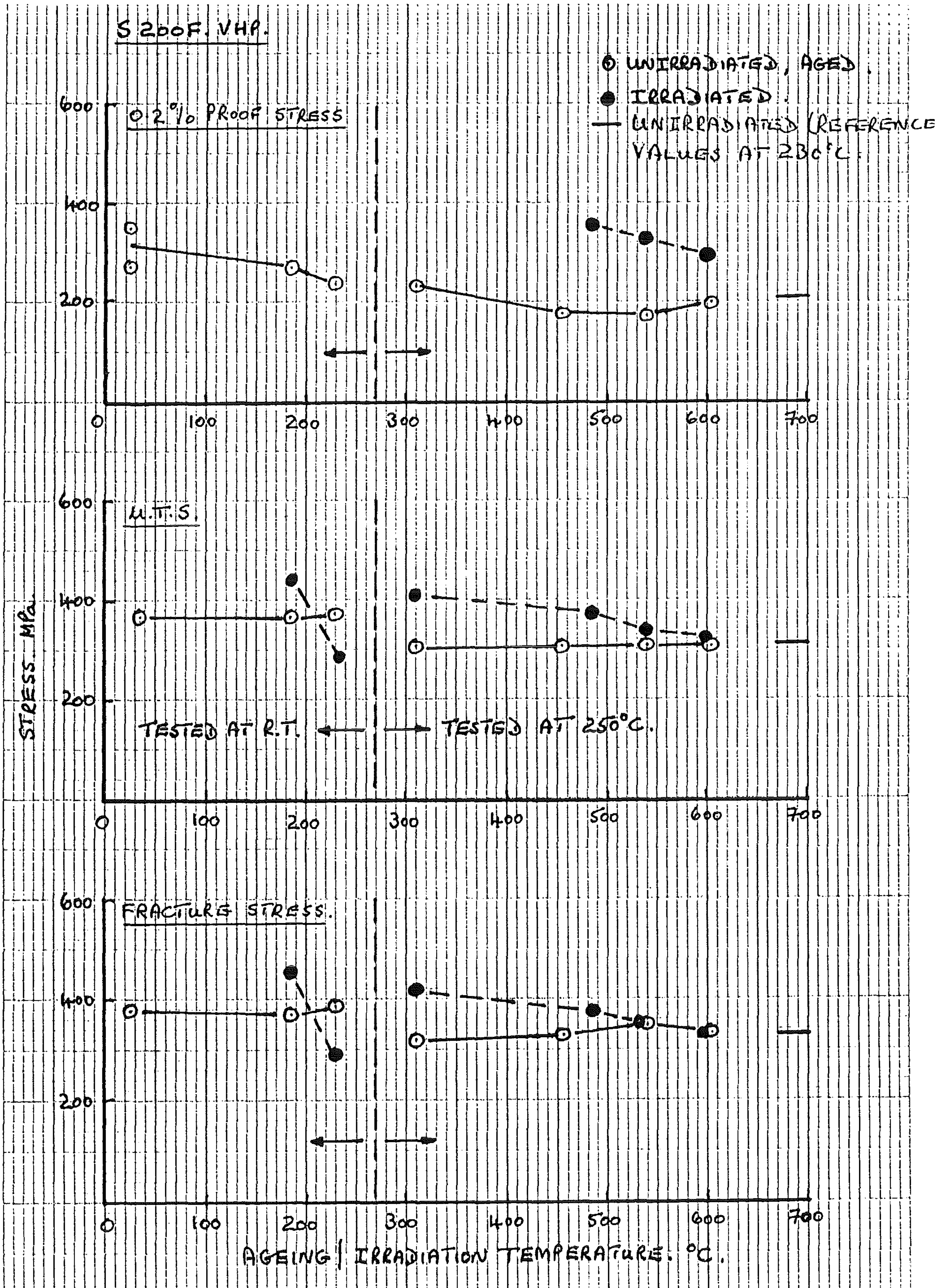
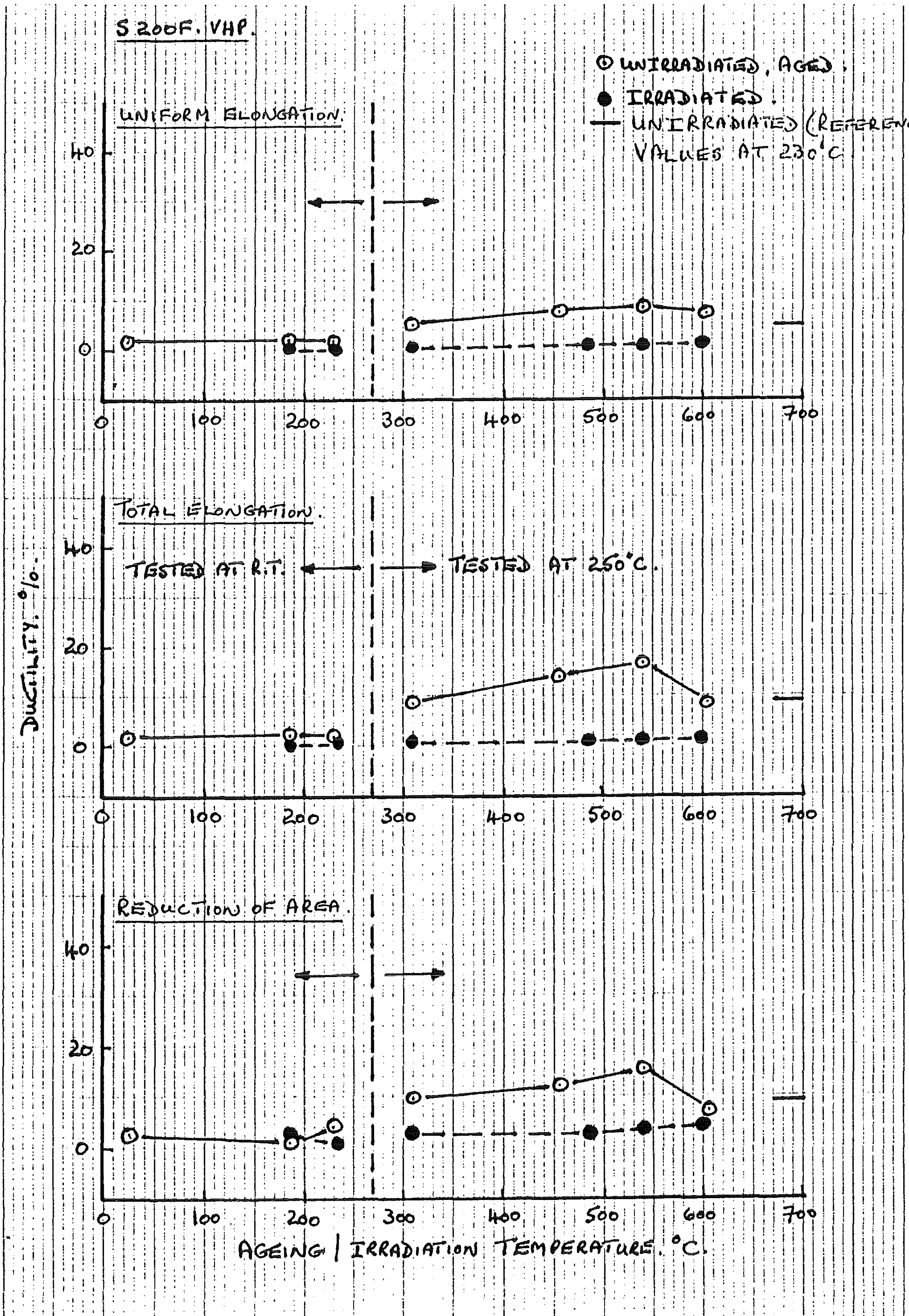

S 200F. VHP.
UNIRRADIATED, AGED.
IRRADIATED.
UNIRRADIATED (REFERENCE VALUES AT 23°C).
0.2% PROOF STRESS
U.T.S.
FRACTURE STRESS.
STRESS. MPa
TESTED AT R.T.
TESTED AT 250°C.
600
400
200
0
100
200
300
400
500
600
700
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. B4

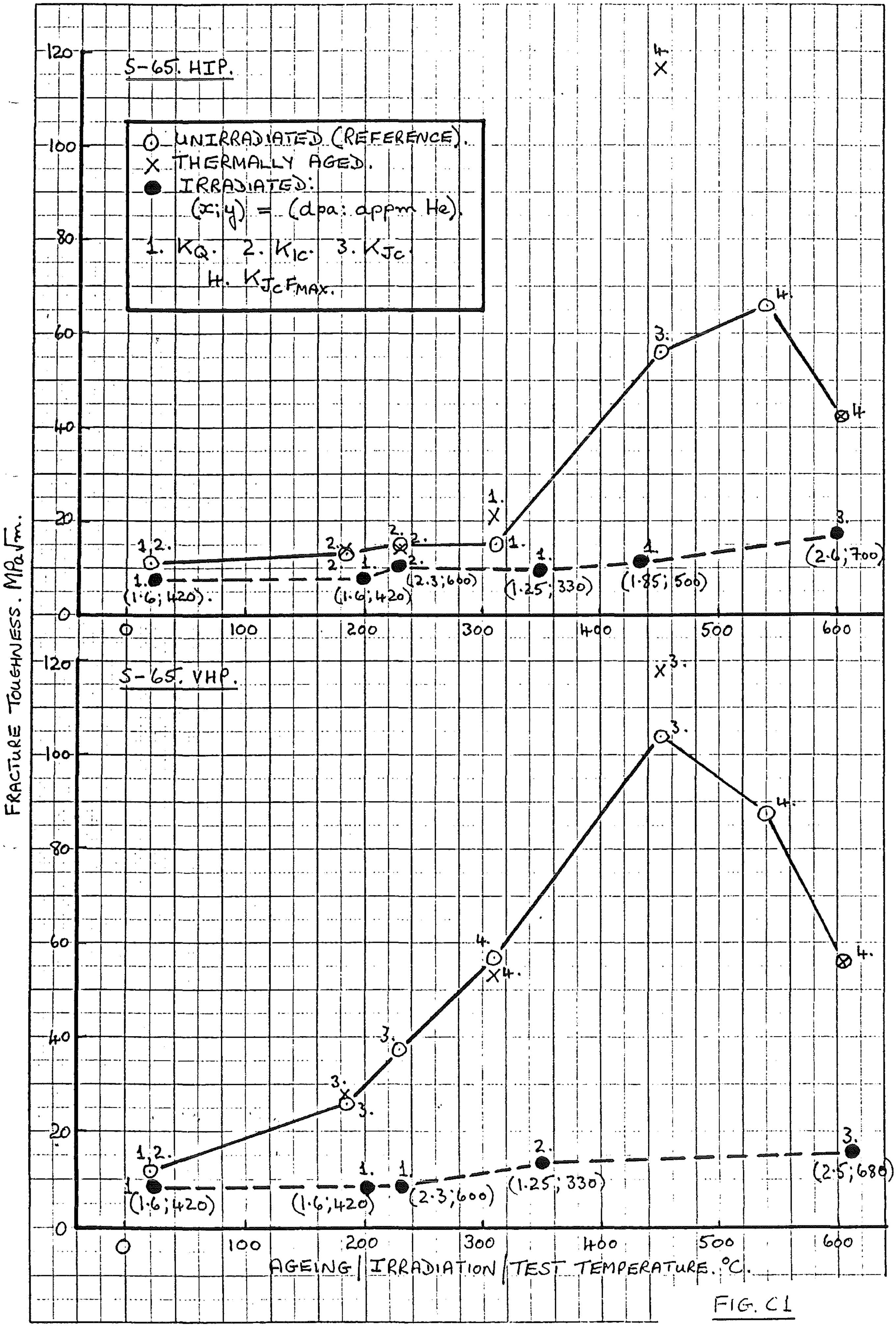


S 200F. VHP.
⊙ UNIRRADIATED, AGED.
● IRRADIATED.
— UNIRRADIATED (REFERENCE) VALUES AT 230°C
UNIFORM ELONGATION.
TOTAL ELONGATION.
TESTED AT R.T.
TESTED AT 250°C.
REDUCTION OF AREA.
DUCTILITY. %.
0
20
40
100
200
300
400
500
600
700
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. B4 (CONTD.)

Appendix C

Effects of Ageing, Irradiation and Test Temperature on the Fracture Toughness of S-65. HIP and S-65. VHP (Fig. C1) and S-200F. HIP and S-200F. VHP (Fig. C2) Beryllium.



S-65. HIP.
UNIRRADIATED (REFERENCE).
X THERMALLY AGED.
IRRADIATED:
(x;y) = (dpa: appm He).
1. KQ. 2. KIc. 3. KJc.
4. KJcFMAX.
(1·6;420).
(1·6;420)
(2.3;600)
(1.25;330)
(1·85;500)
(2·6;700)
S-65. VHP.
(1·6;420)
(1·6;420)
(2·3;600)
(1.25;330)
(2·5;680)
FRACTURE TOUGHNESS. MPa√m.
AGEING | IRRADIATION | TEST TEMPERATURE. °C.

FIG. C1

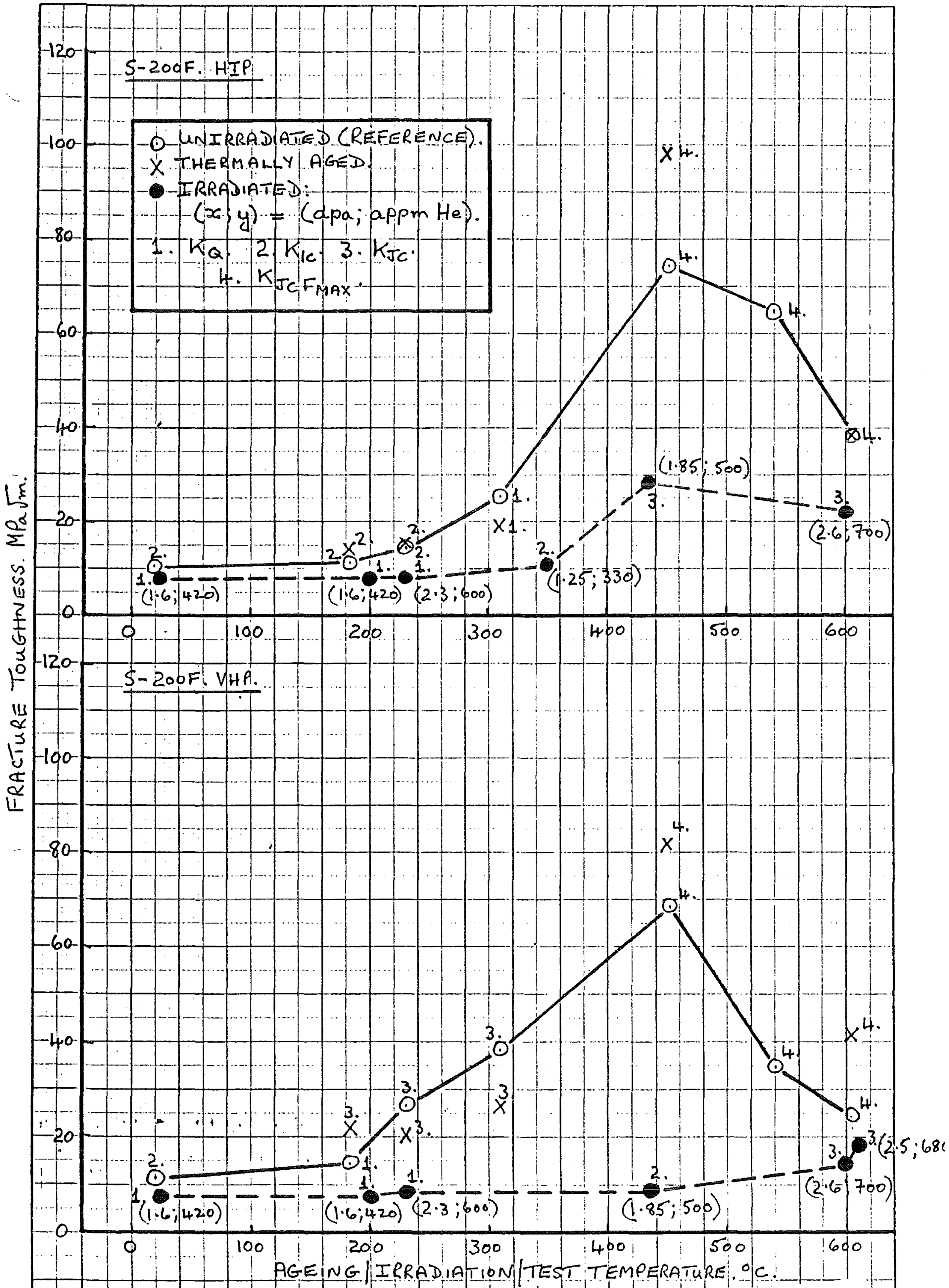

S-200F. HIP
⊙ UNIRRADIATED (REFERENCE).
X THERMALLY AGED.
● IRRADIATED:
(x; y) = (dpa; appm He).
1. KQ. 2. KIc. 3. KJc.
4. KJcFMAX.
(1·6; 420)
(1·6; 420)
(2·3; 600)
(1·25; 330)
(1·85; 500)
(2·6; 700)
S-200F. VHP.
(1·6; 420)
(1·6; 420)
(2·3; 600)
(1·85; 500)
(2·6; 700)
(2·5; 68(
FRACTURE TOUGHNESS. MPa√m.
AGEING | IRRADIATION | TEST TEMPERATURE. °C.

FIG. C2

Appendix D

Effects of Ageing at 185 and 235°C and Irradiation at 200 and 235°C on the Ambient Temperature Fracture Toughness and Ageing at 310, 455, 540 and 605 C and Irradiation at 310, 435, 600 and 610 C on the Fracture Toughness at 250°C of the S-65. HIP, S-65. VHP, S-200F. HIP and S-200F. VHP Beryllium (Fig. D1).

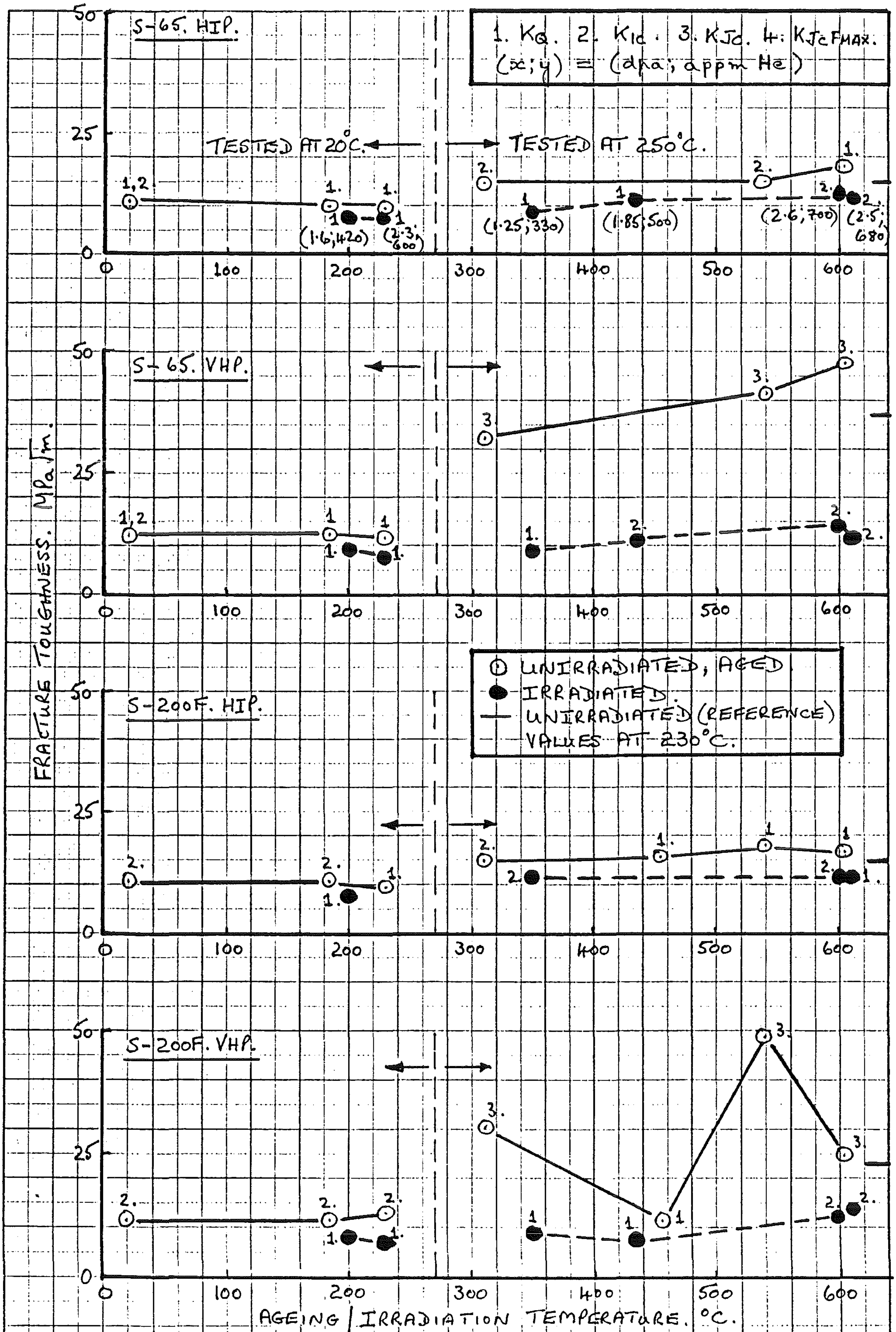

S-65. HIP.
1. KQ. 2. KIc. 3. KJc. 4: KJc FMAX.
(x;y) = (dpa; appm He)
TESTED AT 20°C.
TESTED AT 250°C.
(1·6;420)
(2·3;600)
(1·25;330)
(1·85;500)
(2·6;700)
(2·5;680)
S-65. VHP.
FRACTURE TOUGHNESS. MPa √m.
S-200F. HIP.
UNIRRADIATED, AGED
IRRADIATED
UNIRRADIATED (REFERENCE) VALUES AT 230°C.
S-200F. VHP.
AGEING / IRRADIATION TEMPERATURE. °C.

FIG. D1