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MMP4 Thermomechanical Behaviour during Metals Processing

Near Solidus Tests

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ABSTRACT

This report provides details of experiments to obtain near solidus mechanical data on a range of materials at elevated temperatures. These materials included a Copper-Nickel, an Iron and a Nickel alloy. The tests were performed at NPL in a miniature multiproperty apparatus (the ETMT), at the IRC (University of Wales, Swansea) using ASSET (an Advanced Semi-Solid Elongation Tests System) and at USEM (University of Sheffield) in an induction-heated high temperature test rig.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
Head, Centre for Materials Measurement and Technology

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1 INTRODUCTION

An objective of project MMP4, “Thermomechanical Behaviour during Metals Processing”, is to develop a test method for measuring mechanical properties relevant to casting models where materials exist as a mixture of solid and liquid. The IRC, Swansea is developing an Advanced Semi-Solid Elongation Test (ASSET) system [1,2] for this purpose.

Within project MMP4 it was agreed that comparisons of mechanical data in the near solidus region would be useful in interpretation of the elevated temperature properties. Consequently a series of experiments were performed whereby selected materials (a copper-nickel, an iron and a nickel alloy) were tested in the near solidus region using the following equipment:

- ASSET at the IRC, Swansea
 - ETMT at NPL
- ETMT is an NPL-developed miniature multiproperty apparatus [3,4]
An induction-heated test rig, IHTR, at USEM

This report documents progress achieved in attempts to provide the mechanical data and comments on the test systems used in the intercomparison. Some of the tests also provided information on high temperature methods in support of the development of guidelines for testing.

2 MATERIALS

Three alloys were chosen to cover the temperature range 900-1400 °C:

Material	Source
Cu-Ni alloy	Alfred Ellis and Co
CMSX4	Rolls Royce plc
a single crystal Ni-superalloy	
Fe alloy, Eutectoid steel	British Steel (CORUS) Ltd

The Cu-Ni alloy contained about 10% Ni and was provided as a cast block about 200 x 185 x 22 mm in cross section. The CMSX4 single crystal alloy was provided as cast test bars 12.5 mm diameter by 150 mm long, while the Eutectoid steel was supplied as a concast bar about 350 x 250 x 175 mm. All testpieces were manufactured in the nominal longitudinal direction. Chemical analyses for the materials are given in Table 1.

Table 1 - Chemical Analysis, wt%

Eutectoid Steel	Fe Balance	Mn 0.52	C 0.74	Si 0.24	Ni 0.02	P 0.01	S 0.013	Cr 0.018			
Ni alloy, CMSX4	Ni Balance	Co 9.5	Cr 6.4	Al 5.7	W 6.4	Ta 6.5	Re 2.92	Ti 1.08	Mo 0.7	Hf 0.09	Fe 0.03
Cu-Ni	Cu Balance	Ni 10.27	Mn 0.15	Fe 0.06	Zn 0.17						

3 TESTS

Two sources of information were used to underpin the choice of test temperature:

- IRC performed Thermocalc analyses on the Cu-Ni and the Eutectoid Steel.

NPL used resistivity measurements in the ETMT to indicate the solidus temperature.

The indicated temperatures are given in Table 2.

Table 2 - Indicated Temperatures, °C

Material	Thermocalc (IRC)			Additional Data		ETMT
	Equilibrium Solidus	Scheil Solidus	Liquidus	Solidus	Liquidus	(resistivity data) Solidus
Cu-Ni	1125	1075	1146	1085**	1150**	1060
Eutectoid Steel	1380	1169	1470	-	-	1360 ⁺
CMSX4	-	-	-	1310*	1380*	1350

* provided by Rolls Royce plc

** DSC data-NPL, 10 °C min⁻¹

⁺ It is interesting to note the good agreement between the Solidus value for the eutectoid steel, indicated by the resistivity measurements as 1360 °C, with a value of 1363 °C found by calculation using a thermodynamic solidification model that includes solute diffusion parameters [3].

Figures 1 and 2 show the Thermocalc fraction liquid predictions for the Cu-Ni alloy and the Eutectoid steel. Figs. 3 and 4 show plots of resistance versus temperature for the Cu-Ni alloy and the Ni alloy, CMSX4.

The target temperature ranges for mechanical tests on the three materials were thus chosen to be:

Cu-Ni alloy	1000-1150 °C
Eutectoid Steel	1100-1410 °C
Ni superalloy, CMSX4	1250-1350 °C

The testing strain rates were selected to be in the range 10^{-4} to 10^{-2} s^{-1} .

3.1 TEST SYSTEMS

The NPL and IRC test systems have been described previously [1,2,4,5] but are summarised here together with technical information on the induction-heated USEM test rig, IHTR.

- ASSET

ASSET uses 50 Hz ac current to heat a round testpiece, typically 8-10 mm in diameter, about 160 mm long. Temperature is measured using separately welded thermocouple wires, with the testpiece as the junction. The temperature variation was measured to be about 10 °C within the central 20 mm gauge length in tests on both the Cu-Ni alloy and the Eutectoid steel.

Load is measured using a Hounsfield H10 K-C servo-mechanical test machine with a maximum capacity of 10 kN. The load resolution, using a special low capacity load cell, is about 0.4 N. Strain is measured using a linescan optical system that measures the relative movement of two targets separated by the gauge length of 20 mm. The strain resolution of the linescan system was calculated to be $\pm 0.025\%$, equivalent to a length resolution of $\pm 5 \mu\text{m}$. The test is conducted by applying a constant crosshead displacement rate. The equipment is surrounded by a chamber that allows a controlled test environment to be used for tests on materials that oxidise.

NPL - ETMT

The ETMT uses direct current to heat a rectangular testpiece, nominally 1 x 2 mm in cross section. The testpiece is usually 40 mm long and is held in grips separated by about 16-20 mm. The grips are watercooled and thus there is a temperature distribution along the testpiece when it is heated. At higher temperatures, above 1000 °C, the temperature variation in the central 3 mm was about 5 °C. Force is applied to the testpiece using a computer controlled servomotor with a load resolution of about 0.2N (i.e. about 0.1 MPa stress resolution). Strain is measured using changes in electrical resistance of the central 2-3 mm of the testpiece [5], where the temperature variation is relatively small. The strain resolution is dependent on material resistance but is typically about 0.05%. The test system resides in an environmental box that allows tests to be conducted in flowing argon to minimise oxidation problems. Temperature is measured using spot welded miniature Pt/Pt-13% Rh thermocouples attached to the centre of the testpiece. The test is conducted by applying a constant ramp loading rate.

USEM - IHTR

A Mayes servohydraulic test machine was used for the USEM IHTR tests. The machine has a capacity of 50 kN with a resolution of 2.5 N. It uses an 8 kW induction heater for heating the

testpiece, which is surrounded by a silica tube filled with flowing argon at $10 \text{ litres min}^{-1}$ to prevent oxidation. Displacement is measured out of the hot zone and a nominal gauge length is assumed for calculation of strain. Temperature is measured using a pyrometer sighted on the testpiece centre and with a thermocouple in a hole along the central axis of the testpiece to a depth of 1.5 mm above the gauge section. The testpiece is circular and is 120 mm long and 6.5 mm in diameter in the centre gauge length. The hot zone is 65 mm long, and the gauge section is 20 mm long. The temperature in the gauge section has a uniformity of $\pm 5^\circ\text{C}$ and was mapped to the thermocouple temperature using the following procedure. A specimen of the test material was heated to a nominal value of 950°C . A pyrometer was then used to measure the temperature at the point of the thermocouple insertion depth and adjusted to the thermocouple reading. A 3°C difference was assumed between the specimen surface temperature and internal centre temperature. The temperature at the centre of the gauge length section was then measured with the pyrometer. A linear relationship was assumed between the thermocouple point and gauge length centre temperature allowing the specimen temperature to be set using the temperature at the thermocouple. A heating curve was then calculated for each temperature at the thermocouple. A heating curve was then calculated for each temperature and the specimen heated for 600s before the start of the test.

Load, displacement and thermocouple temperature data were recorded via the LabTech Notebook Pro. Software package. A maximum sampling rate of 1000 samples/second was achievable.

The load-displacement curves were corrected for machine compliance and converted to engineering stress – engineering strain curves. Subsequent FEM modelling of the tensile test has shown that strain in the gauge section is not homogeneous and up to 20% of the displacement of the specimen may take place outside the gauge length. This has not been accounted for when converting displacement to engineering strain.

4 RESULTS

4. Cu-Ni ALLOY

The Cu-Ni alloy was tested in both the ETMT and ASSET. Typical stress strain curves are shown in Figs. 5 and 6. In some of the ETMT tests the testpiece was deformed in compression as well as tension. The tension data from the ETMT and ASSET are compared in Fig. 7. It can be seen that the effects of strain rate are very significant. The strain rates are average values for each test. The ETMT tests were load controlled while the ASSET tests were based on crosshead control.

The ETMT and ASSET strength values plotted in Fig 7 were obtained at strains of several % where the stress/strain curves were close to or had reached a saturation value of stress.

The high conductivity of the Cu-Ni alloy meant that it was not possible to heat the testpieces in the USEM IHTR rig.

4.2 Ni ALLOY, CMSX4

Tests on the CMSX4 alloy were performed in the ETMT and the IHTR.

Typical tension and compression stress/strain plots obtained using the ETMT are shown in Figs. 8-10. A summary plot of strength data obtained at a strain of 1% is shown in Fig 11. Again, as for tests on the Cu-Ni alloy a strong effect of strain rate can be seen.

Tests were carried out using the USEM IHTR Mayes machine with induction heating to nominal temperatures of 1250 °C, 1280 °C, 1310 °C, 1330 °C, 1350 °C and strain rates of 0.01 s⁻¹ and 0.001 s⁻¹.

The IHTR specimens had the holes to take the thermocouples spark eroded because of problems with machining. Slight differences (± 1 mm in some cases) in the depth of the thermocouple hole has been found to cause differences in the temperature in the gauge length of the specimen due to the temperature map being calculated for a specific depth. These differences clearly alter the gauge length temperature quite dramatically and possibly account for the apparent discrepancies in load values with respect to temperature, especially in the lower strain rate tests. These tests were performed after the higher strain rate tests and the 'more accurate' specimens were chosen for the latter. Also because several specimens had to be used to refine the temperature map and a couple of tests had to be repeated due to melting of the specimens it was not possible to complete the lower strain rate tests with the temperature at 1350 °C. Figures 12 and 13 show the stress-strain data.

Stress-strain curves in the higher strain rate tests show a rapid rise to a maximum then possibly a region of steady state behaviour followed by a decrease to failure. In contrast the lower strain rate tests show a rapid rise in stress followed by a region of steady state then a gradual rise in stress from a strain of approximately 0.05 followed by a decrease to failure.

Figure 14 shows a plot of stress (at a strain of 0.05) against inverse temperature for the CMSX4 alloy (IHTR data) with scatter arising, it is thought, from uncertainties in temperature. In particular the data at 1245 °C and a strain rate of 0.001 s⁻¹ is considerably in error. A true test temperature in this case is indicated as 1315 °C using Fig 14.

For comparison the ETMT data is also plotted against inverse temperature in Fig 15. The two sets of data are not easily comparable because of the different strain rates and it is clear that at these high temperatures strain rate is a significant testing parameter. However, the IHTR tests seem to be yielding higher flow stresses than the ETMT and this discrepancy remains to be resolved.

4.3 EUTECTOID STEEL

Tests on the Eutectoid steel were carried out in the ETMT and the IHTR. Also, one test was reported from ASSET at a temperature of approximately 1400 °C to indicate the potential of the system (Fig 16).

Typical stress/strain plots from the ETMT and the IHTR are shown in Figs 17-22. The IHTR tests reached a peak value of stress before necking of the testpiece intruded on the measurement process. The matrix of tests carried out using the Mayes IHTR system used induction heating to nominal temperatures of 1100 °C, 1200 °C, 1300 °C, 1350 °C, 1370 °C and 1410 °C and strain rates of 0.001 s⁻¹ and 0.01 s⁻¹. The load-displacement curves (Figs 23 and 24) show a rapid rise to a maximum load, possibly followed by oscillating loads before a general decrease in load to failure. Conversion of load to stress assuming homogeneous strain and correcting for machine compliance does not lead to steady state stress. Tests where deformation was stopped after displacements of 5 mm and 10 mm show that necking was evident after 5 mm and well developed after 10 mm (i.e. nominal strains of 0.22 and 0.4). It is considered that the stress conversion is not valid after the first peak, because of initiation of necking. The test result at 1450 °C and 0.01 s⁻¹ indicates that partial melting had occurred because no load could be recorded. Fig 25 shows the IHTR peak strength data plotted against inverse temperatures. These peak values are compared with the ETMT and ASSET data in Fig 26. Clearly, there are significant differences that can only be in part explained by differences in strain rate.

5 MICROSCOPY

A Cu-Ni testpiece was heated to melting in the ETMT. On melting the testpiece separated. This resulted in a quench of both remaining halves of the testpiece. These testpiece halves were sectioned and polished. Figure 23 shows the dendritic structure in the unmelted region where the temperatures were below the solidus. Figure 24 shows the refined dendritic structure in the melted region in the centre of the testpiece and also the interface between the unmelted and melted regions. This region is shown at a higher magnification in Fig. 25. These micrographs illustrate the importance for modelling of not only knowing how much liquid is present but also what is the morphology of the solid/liquid structure in any given test material.

6 SUMMARY

The planned test matrix was not fully completed because of problems with developing the test systems to obtain the required data at elevated temperatures. For example:

- No ETMT tests were conducted in the solid/liquid region because with the present software control it was found difficult to hold the temperature constant once liquid formed. A faster computer would be needed, with at least a factor 10 improvement in speed.
- No ASSET tests were conducted on the Eutectoid steel and the Ni alloy because the strain measurement system was not fully developed for use at temperatures above 1100 °C within the time frame of the project. Also some difficulties were observed with temperature control of the Eutectoid steel during ramping to the test temperature through the phase transformation.

- No IHTR tests were conducted on the Cu-Ni alloy because it proved difficult to heat due to its low resistivity. Also, because of the need to assume a gauge length in the hot zone, strain values from this system are nominal values only.

Only a limited comparison of data from the different systems was possible because of limitations in test procedures for all three test systems. However, each system appeared to give self-consistent results.

The high temperature measurement requirement is for stress/strain curves at specified temperatures and deformation rates in the near solidus region. Tests in the current exercise have shown that with some further limited development both ETMT and ASSET could probably be used for this purpose. The ETMT can provide stress/strain curves up to the solidus but needs a faster computer for improved temperature control beyond the solidus and requires modification for constant strain rate as opposed to constant stress rate tests. It would also need to be confirmed that the small cross-section is representative of the cast microstructures and that resistance changes are still consistent with strain changes in the presence of a liquid. Failing this a linescan system would be needed to measure distance (strain) between two targets on the testpiece. ASSET requires further development of the linescan system to obtain strain data in tests at temperatures above 1100 °C and suitable heating profiles would need to be developed, particularly for ferrous materials. The IHTR equipment requires considerable further modification to allow direct unambiguous measurement of strains and to ensure reasonably uniform temperatures in the region of interest.

It is clear from the variation in temperatures indicated in Table 2 that measurements of mechanical properties would also be enhanced by a knowledge of the morphology and volume fraction liquid in tests on materials in the solid/liquid region.

7 ACKNOWLEDGEMENTS

The report summarises input from the MMP4 Consortium members at NPL, IRC (University of Wales, Swansea) - Dr J James and colleagues and USEM (University of Sheffield, Engineering Materials) - Prof C M Sellars and colleagues. The project was funded by the DTI within the "Materials Measurements which affect Processability" programme.

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 Eutectoid steel - Thermocalc predictions.
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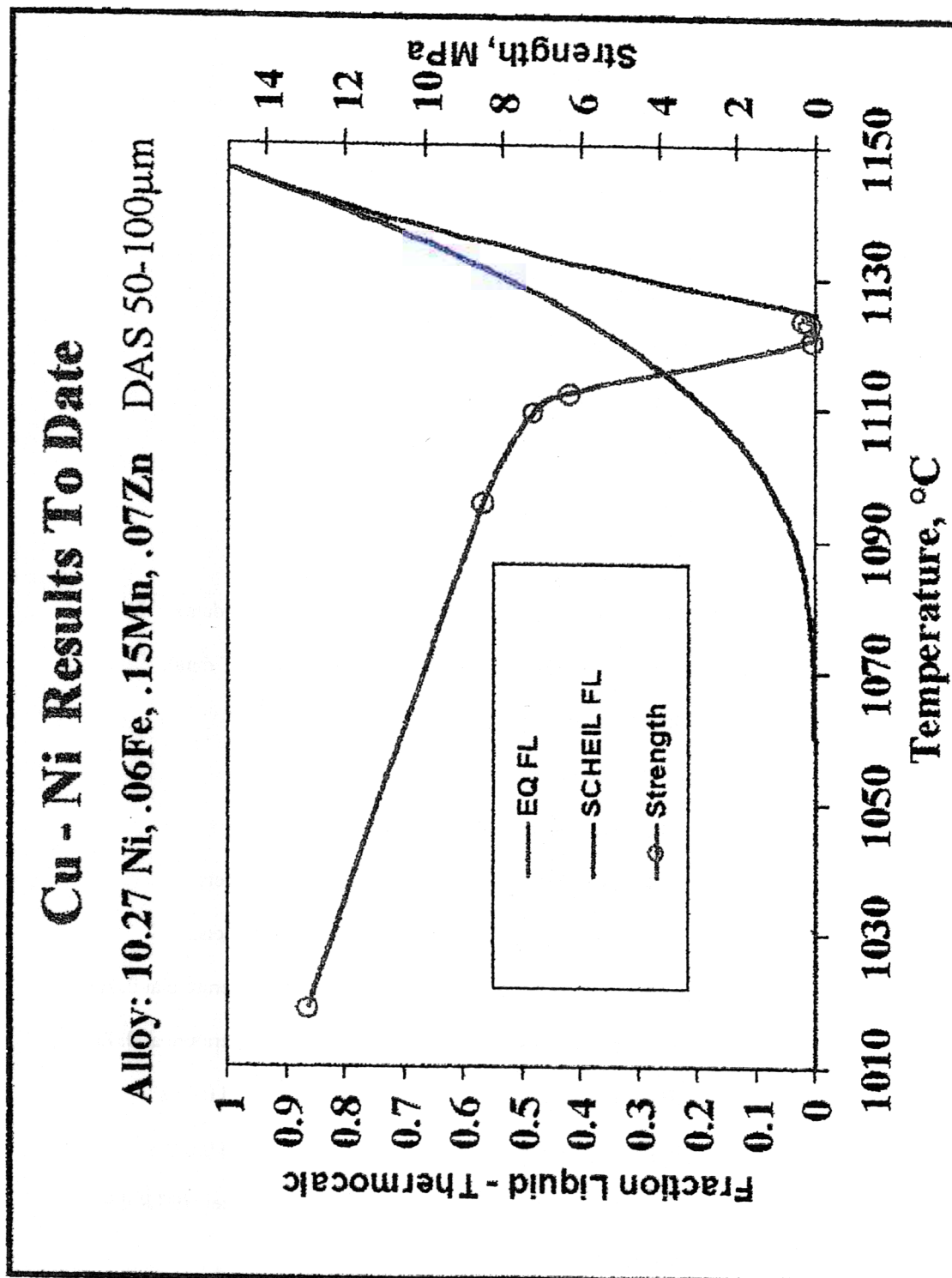


Fig 1 Cu-Ni alloy - Thermocalc predictions.

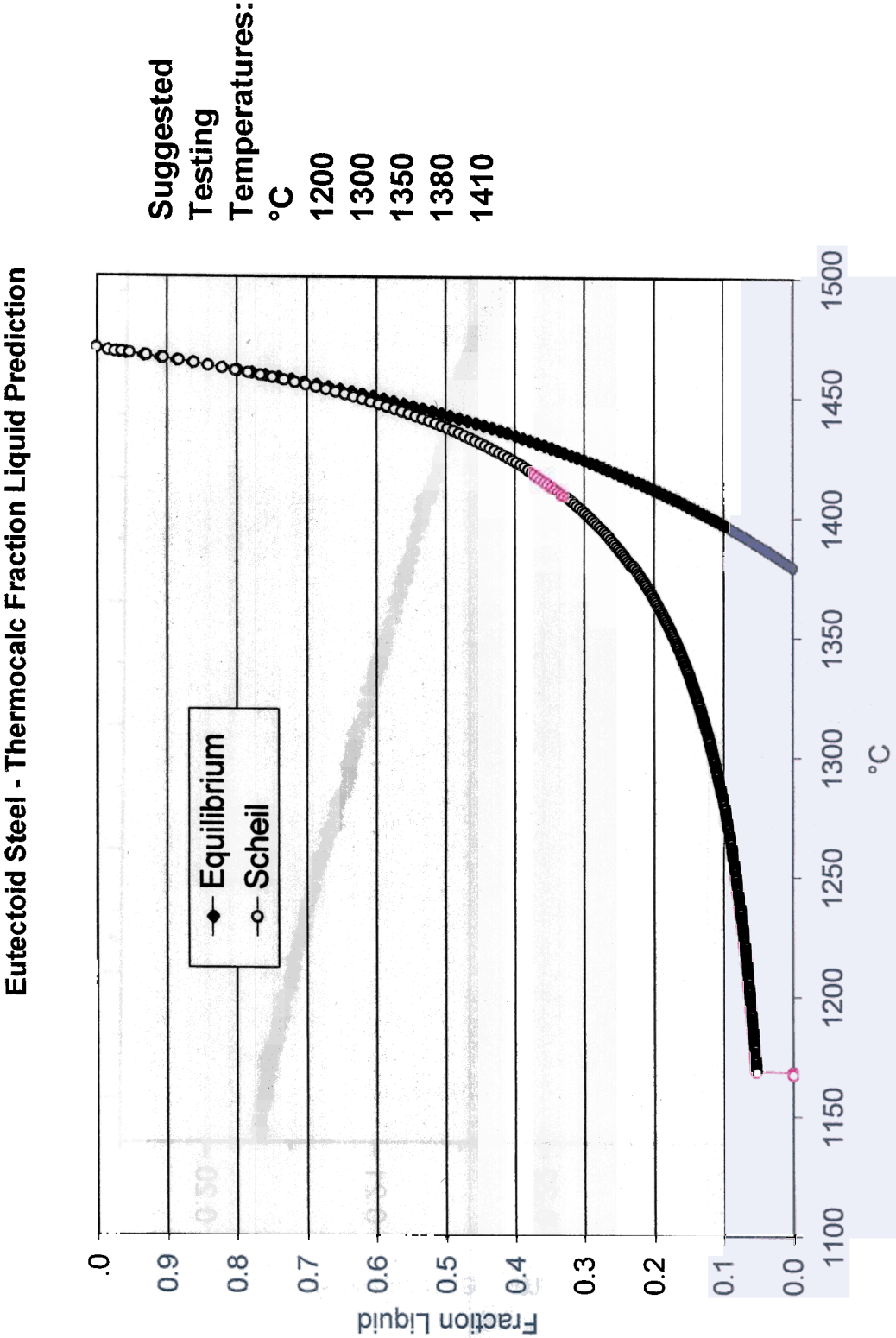


Fig 2

Eutectoid steel - Thermocalc predictions.

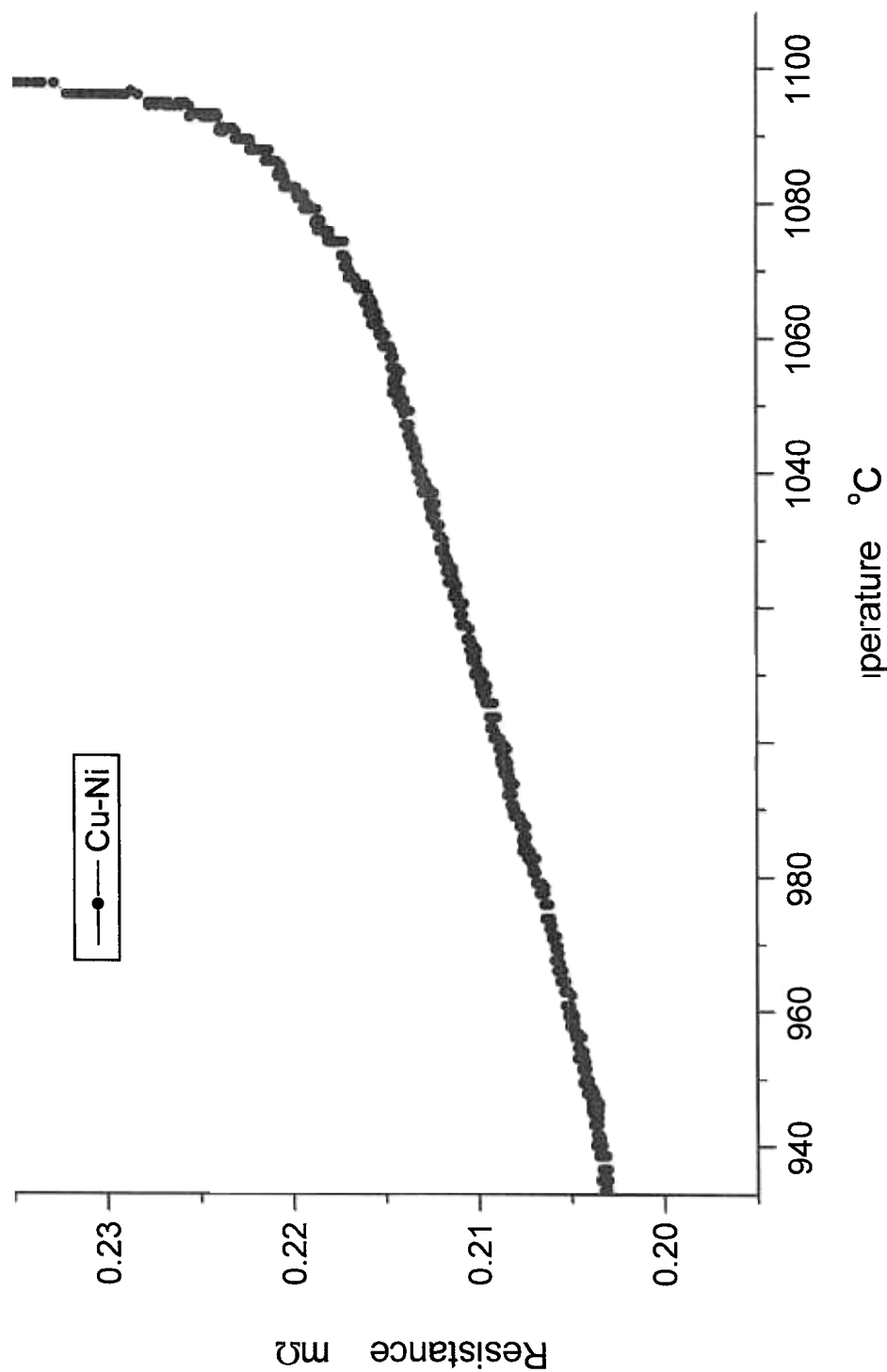


Fig 3 Resistance versus temperature for Cu-Ni alloy.

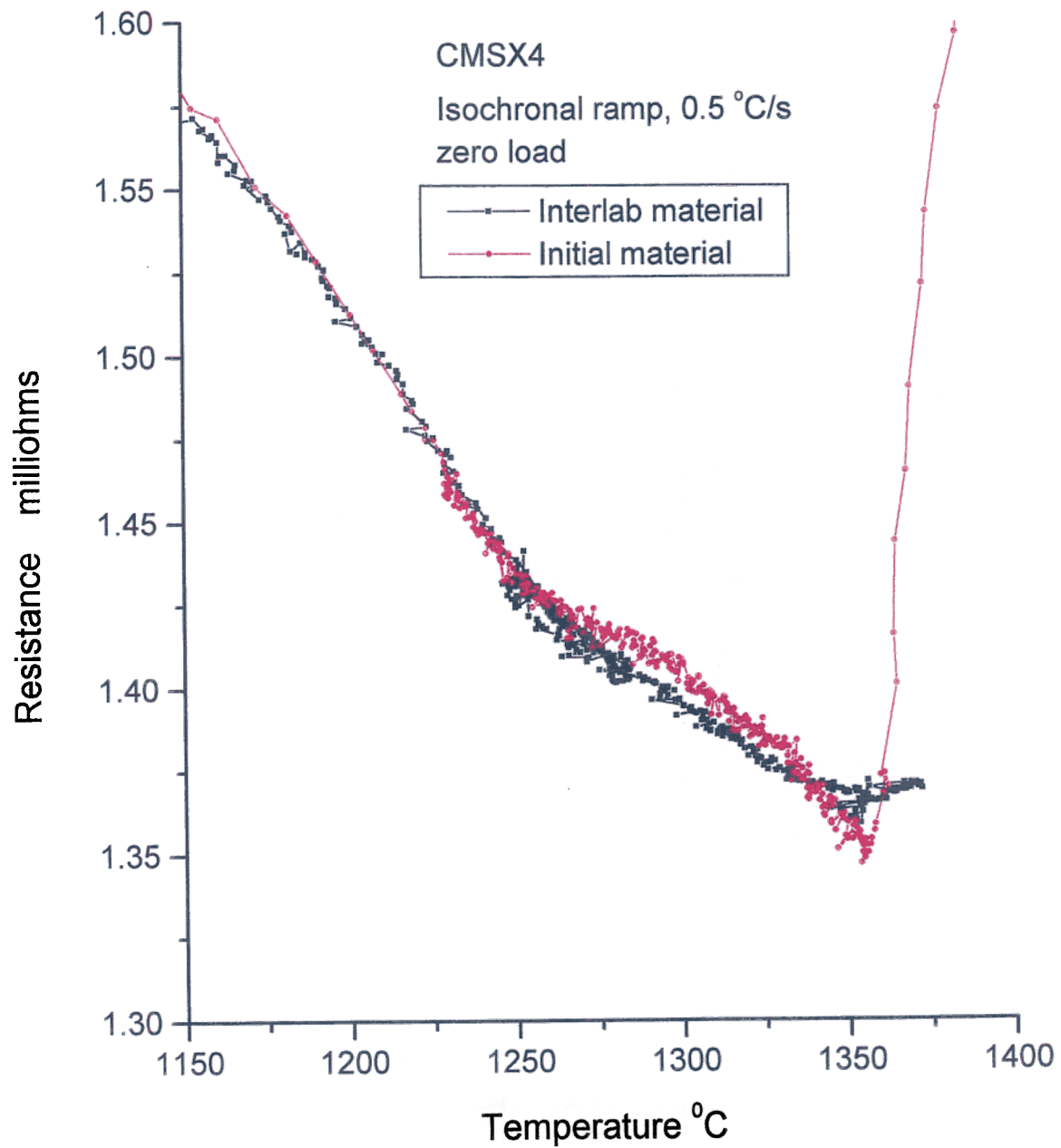


Fig 4 Resistance versus temperature for Ni alloy, CMSX4.

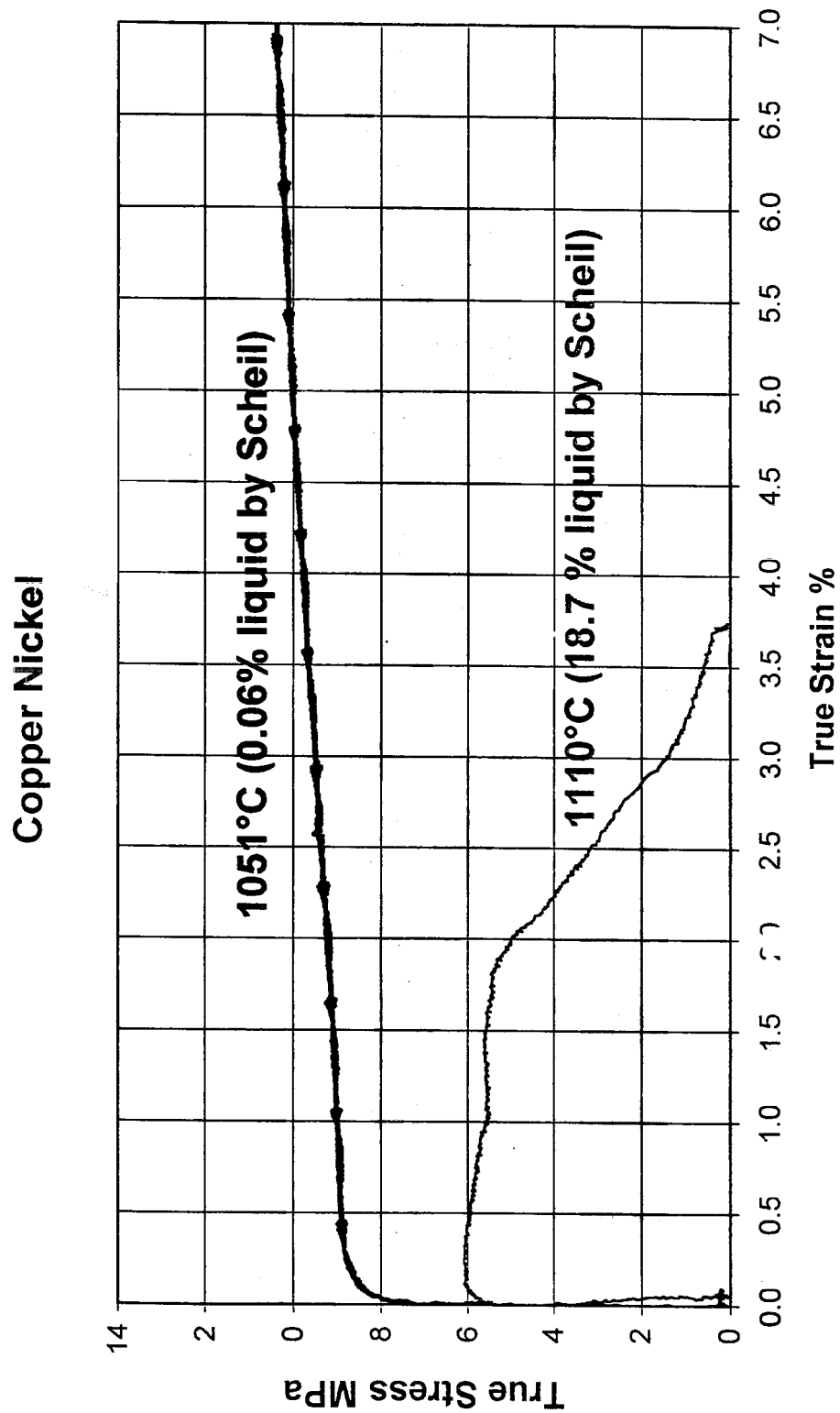


Fig 5 ASSET stress/strain curves on Cu-Ni alloy.

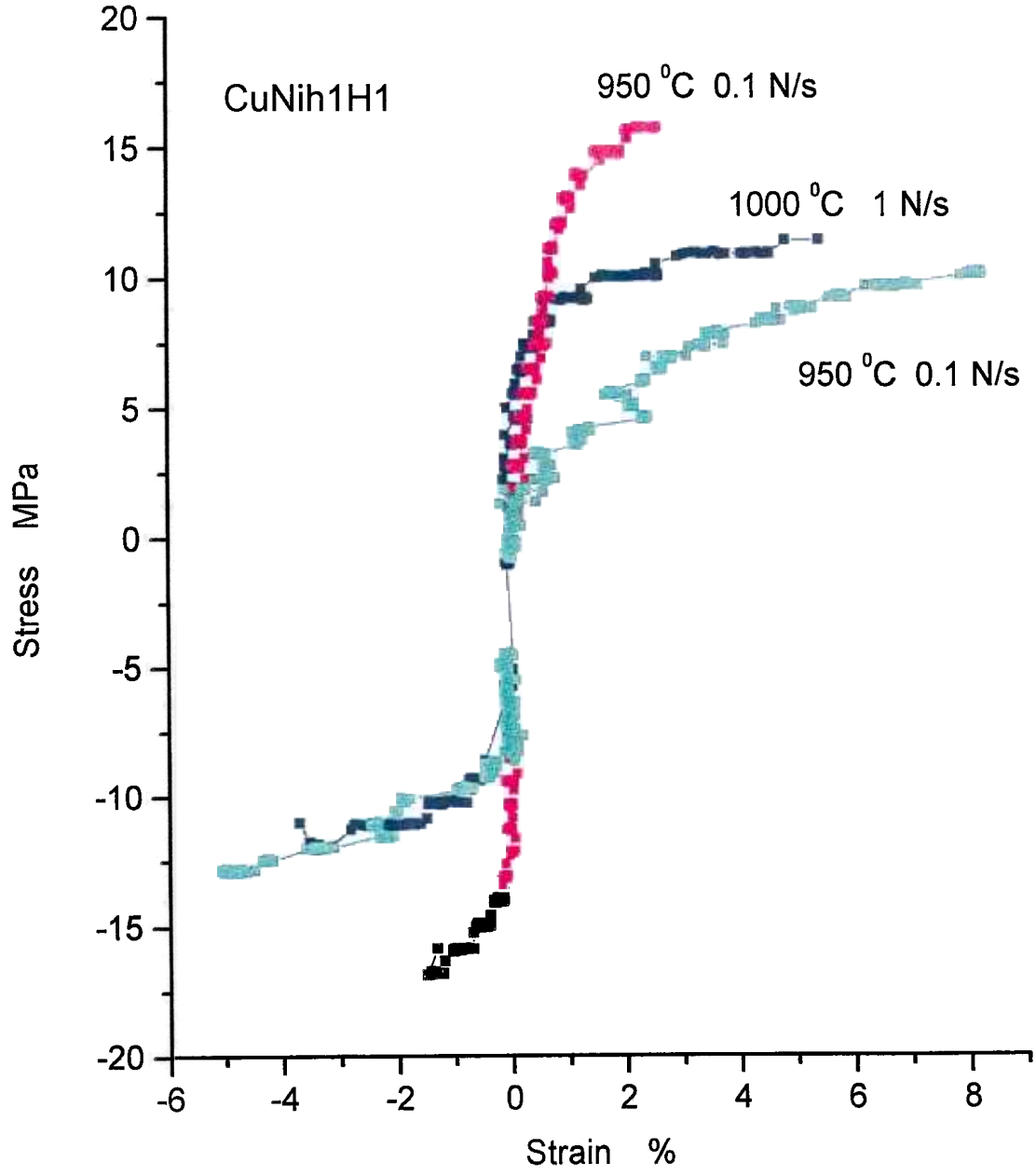


Fig 6 ETMT stress/strain curves on Cu-Ni alloy.

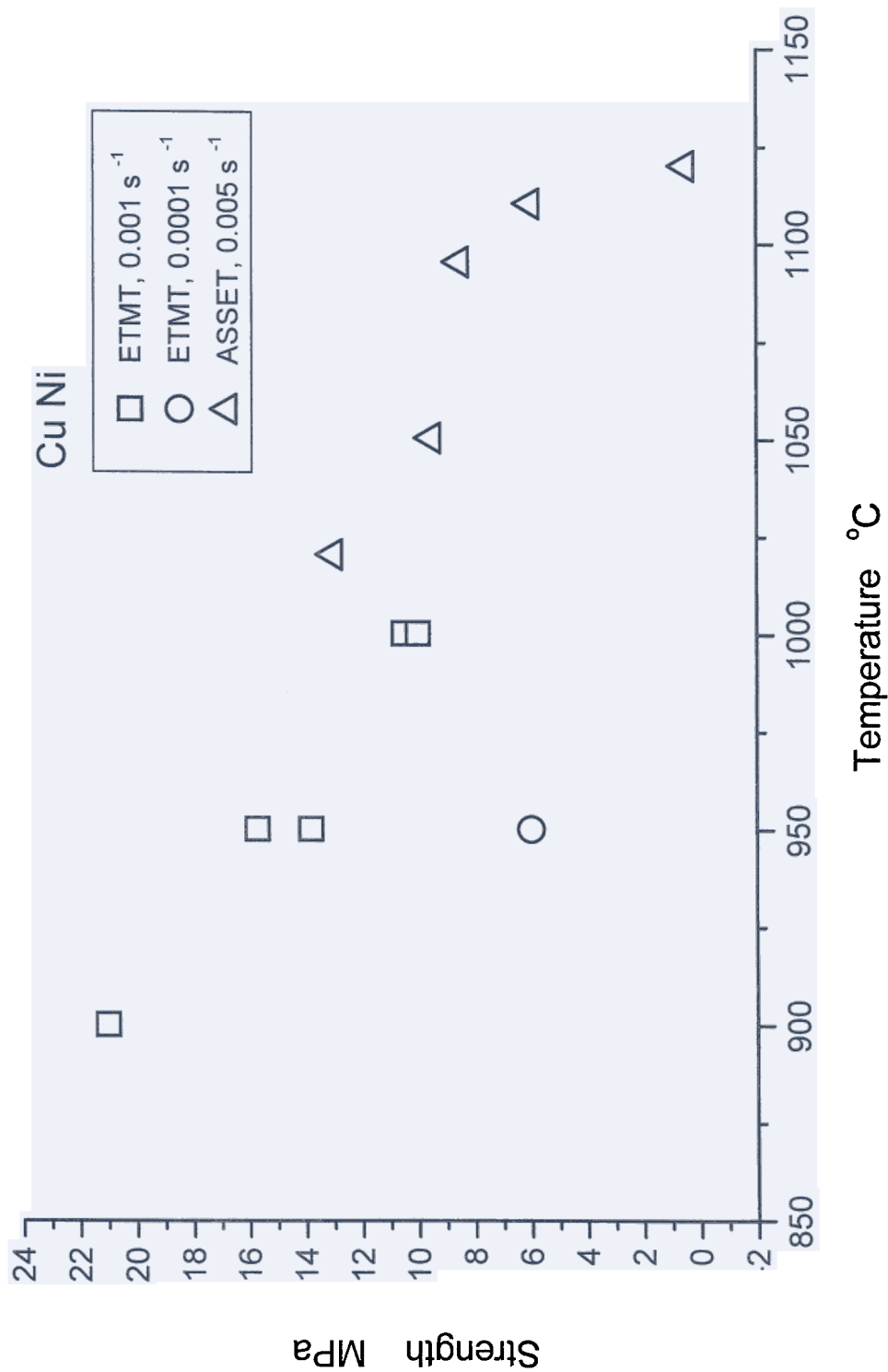


Fig 7 Comparison of Cu-Ni strength values

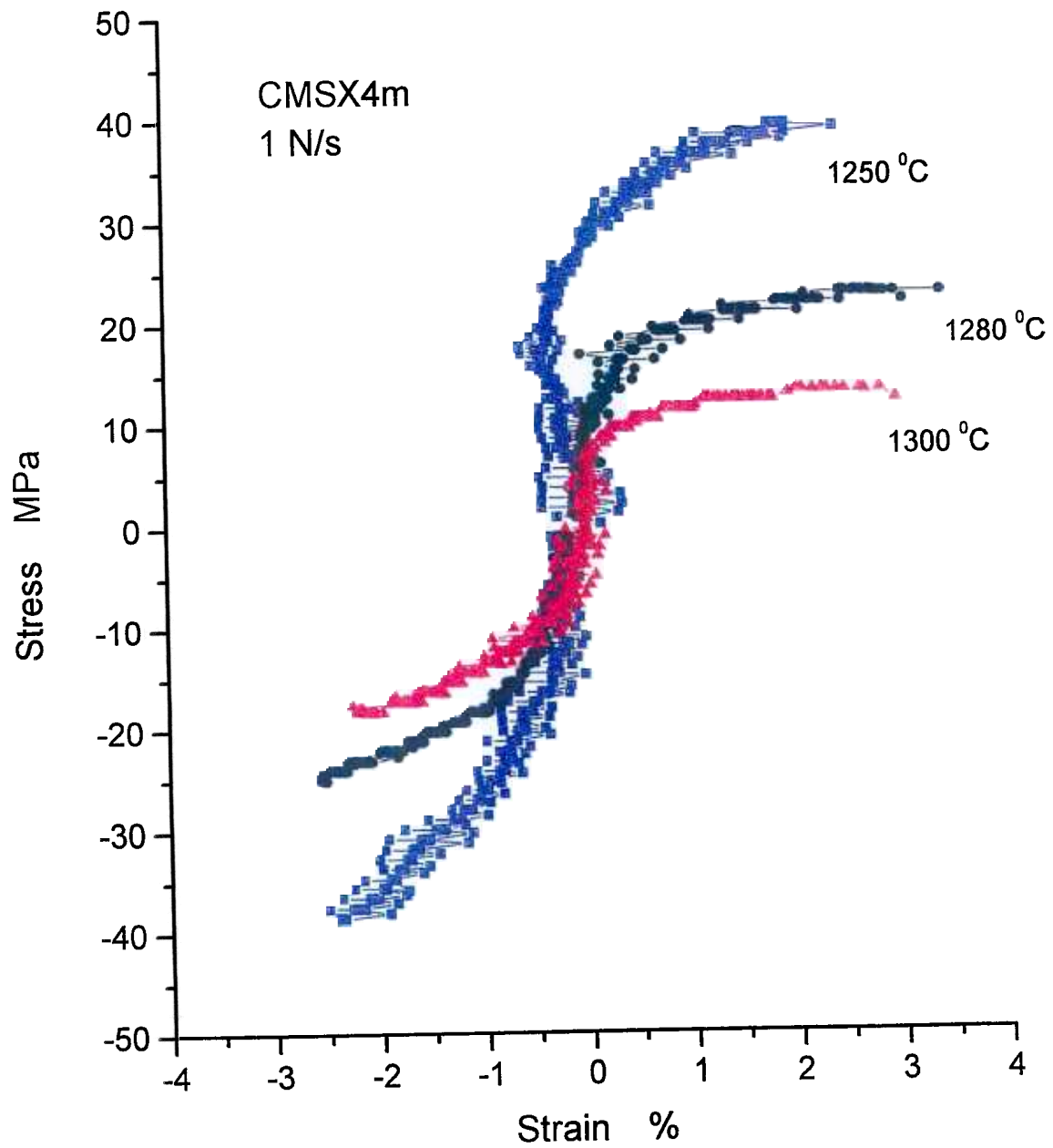


Fig 8 ETMT stress/strain curves on CMSX4 at 1 Ns⁻¹ loading rate.

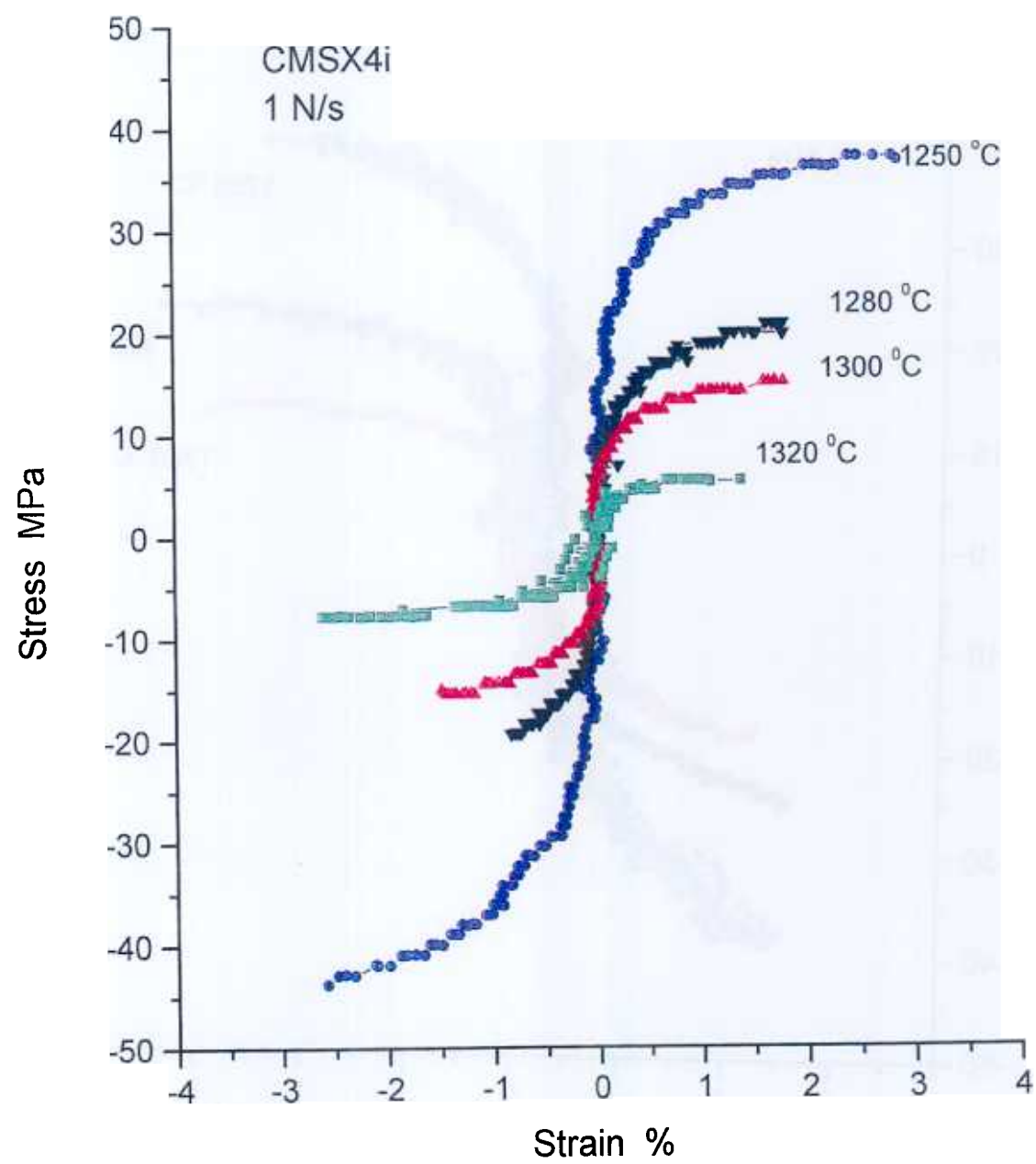


Fig 9 Additional ETMT stress/strain curves on CMSX4 at 1 Ns⁻¹ loading rate.

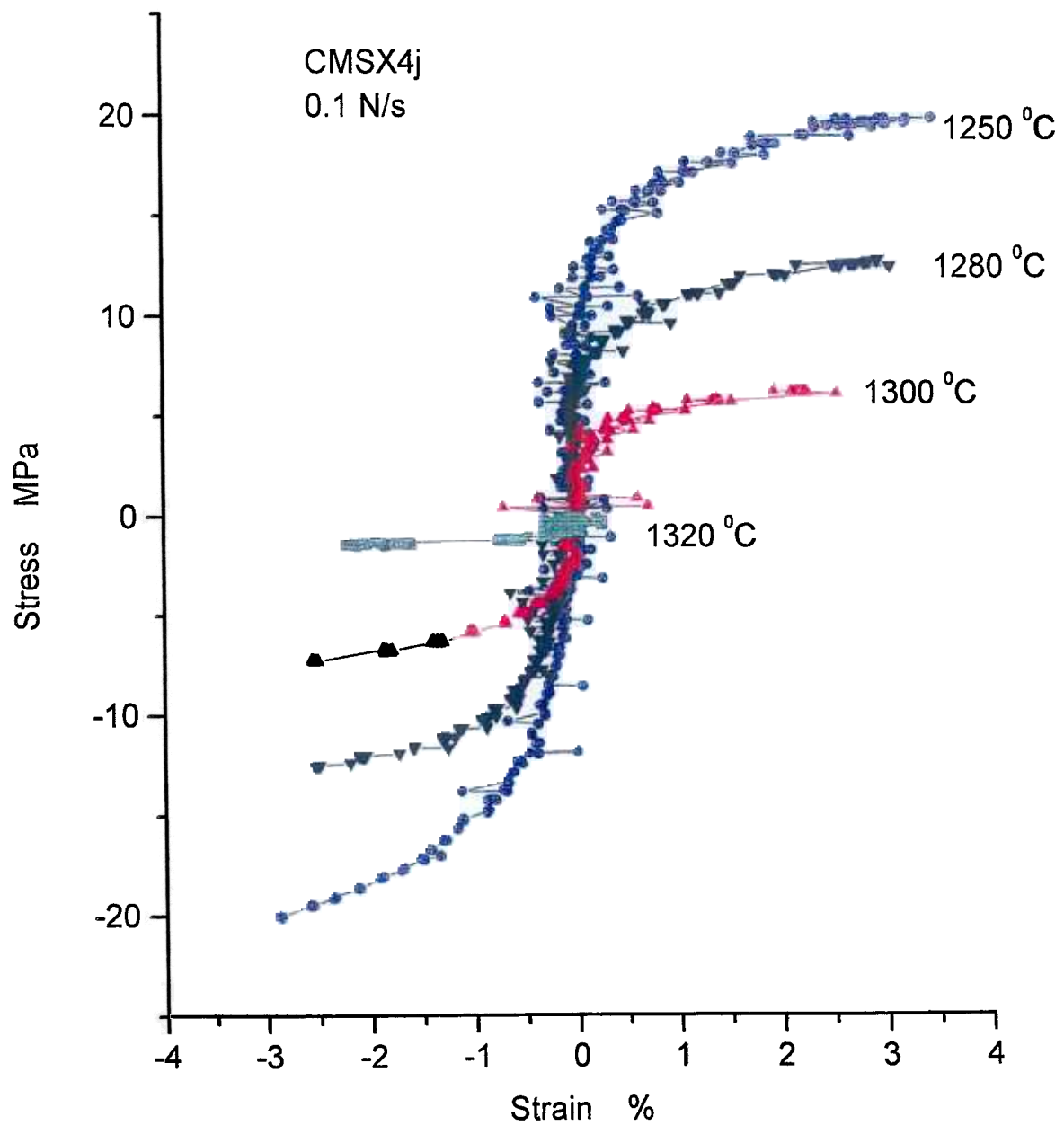


Fig 10 ETMT stress/strain curves on CMSX4 at 0.1 N s^{-1} loading rate.

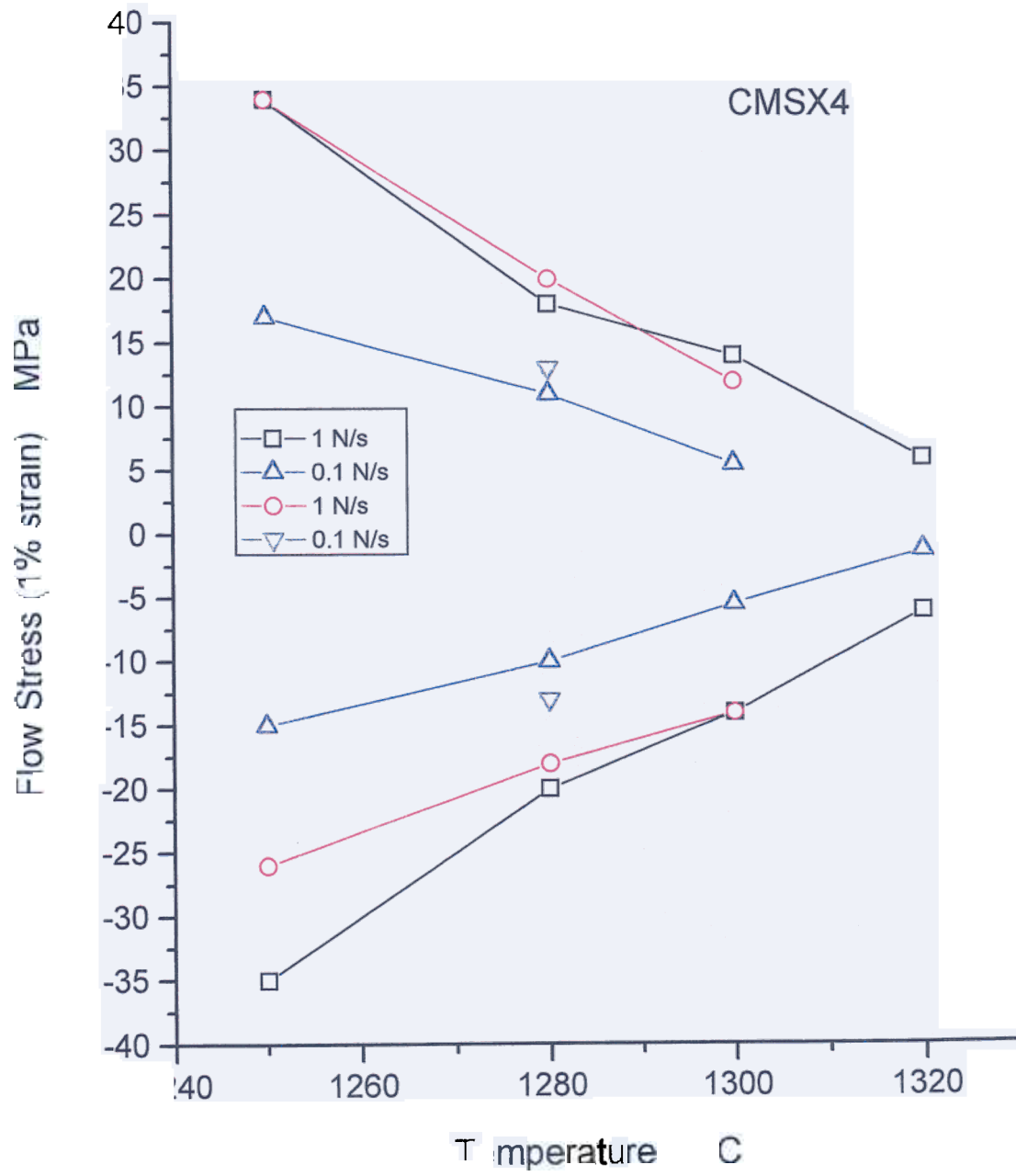


Fig. 1. Flow stress of CMSX4 at 1% strain of (ETMT).

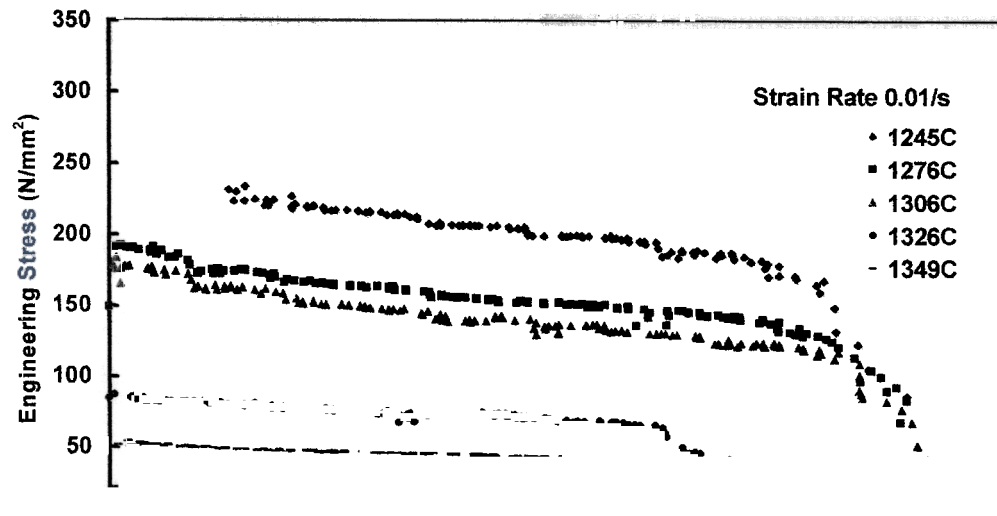


Fig 12 IHTR tests on CMSX4 at 0.01 s^{-1}

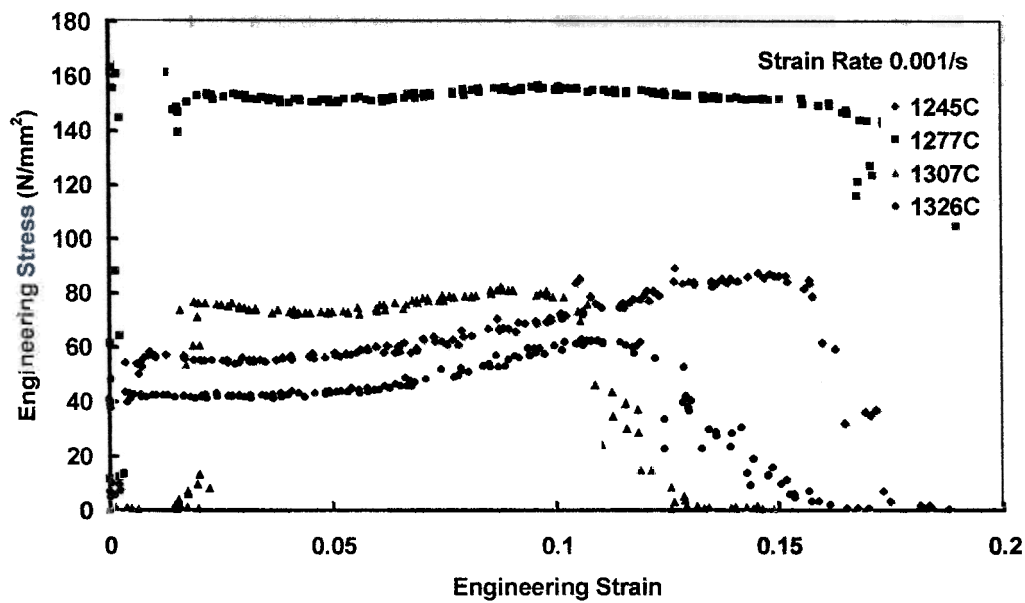


Fig 13 IHTR tests on CMSX4 at 0.001 s^{-1}

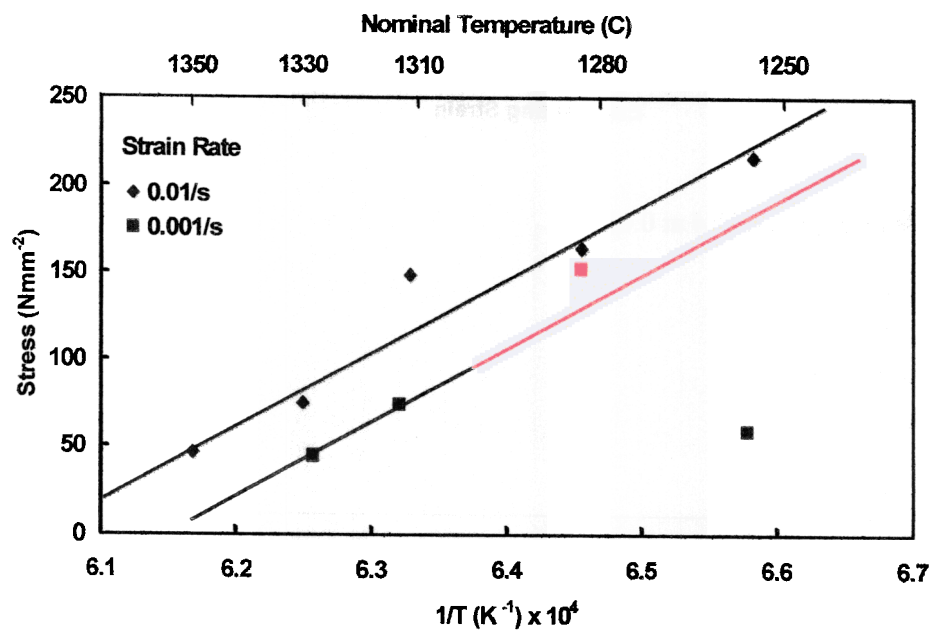


Fig 14 Plot of stress at a strain of 0.05 against inverse temperature for CMSX4 (IHTR data)

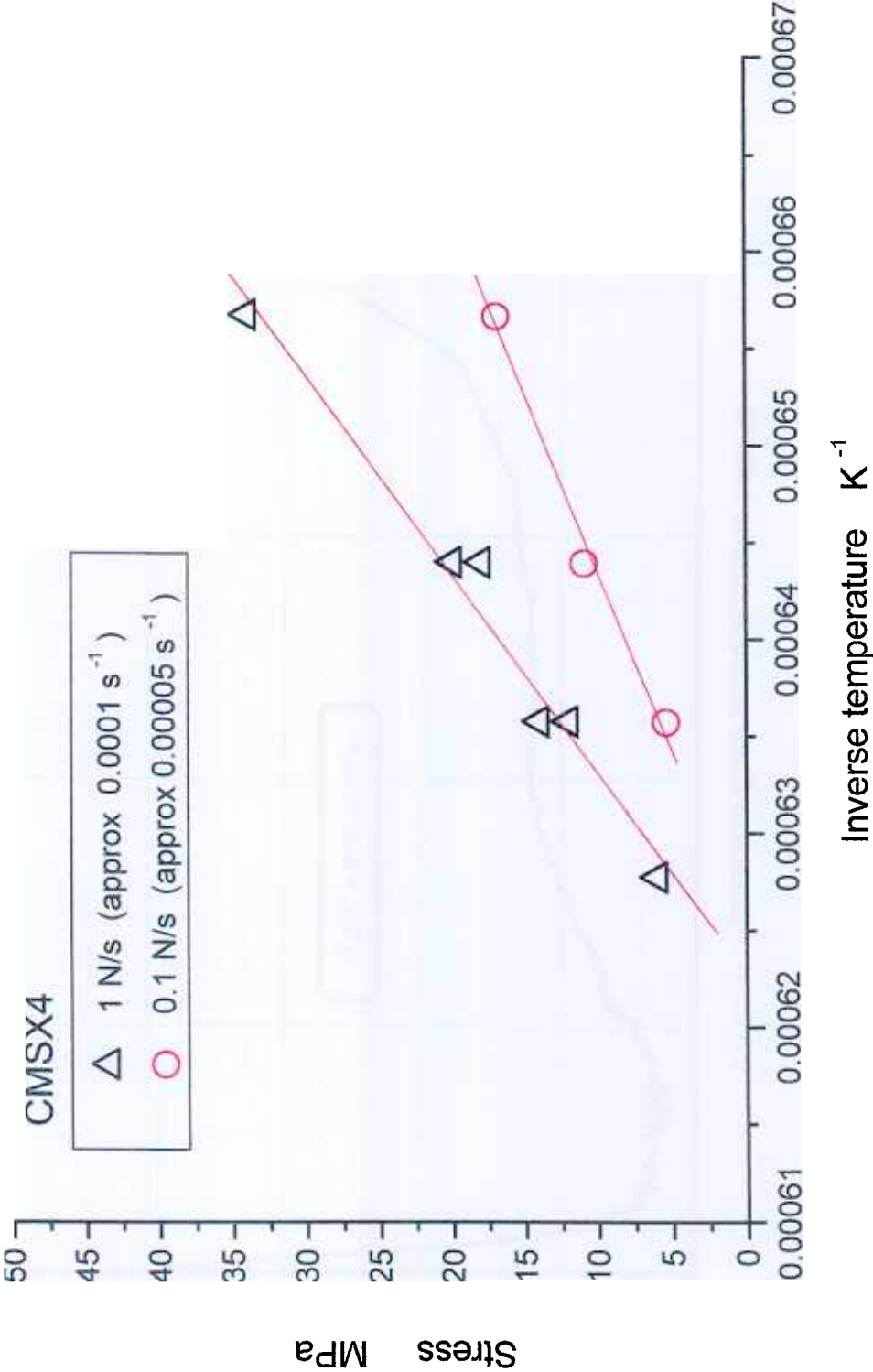


Fig 15 Plot of stress at a strain of 0.01 against inverse temperature for CMSX4 (ETMT data).

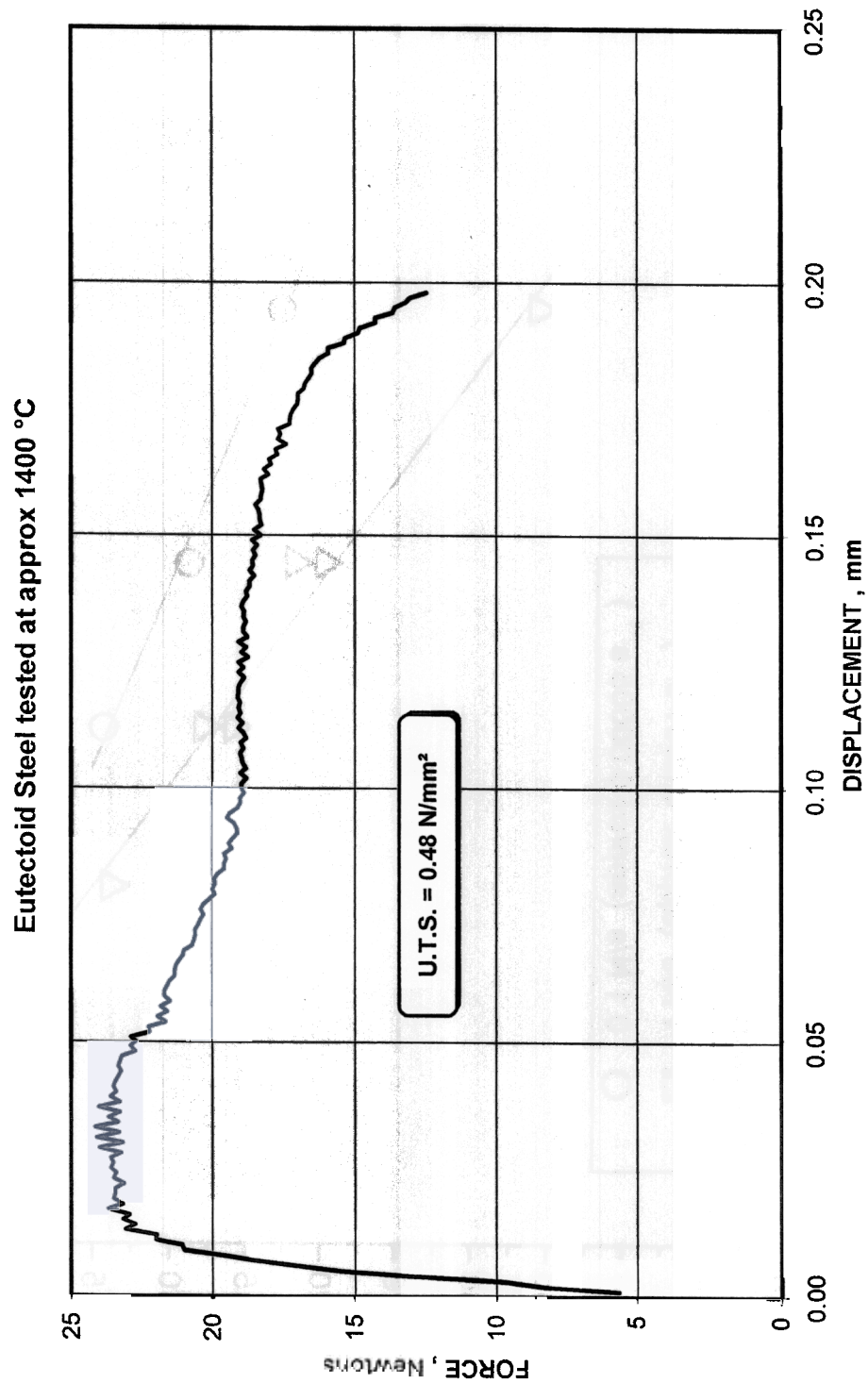


Fig 16 ASSET stress/strain plot at 1400 °C on the Eutectoid steel.

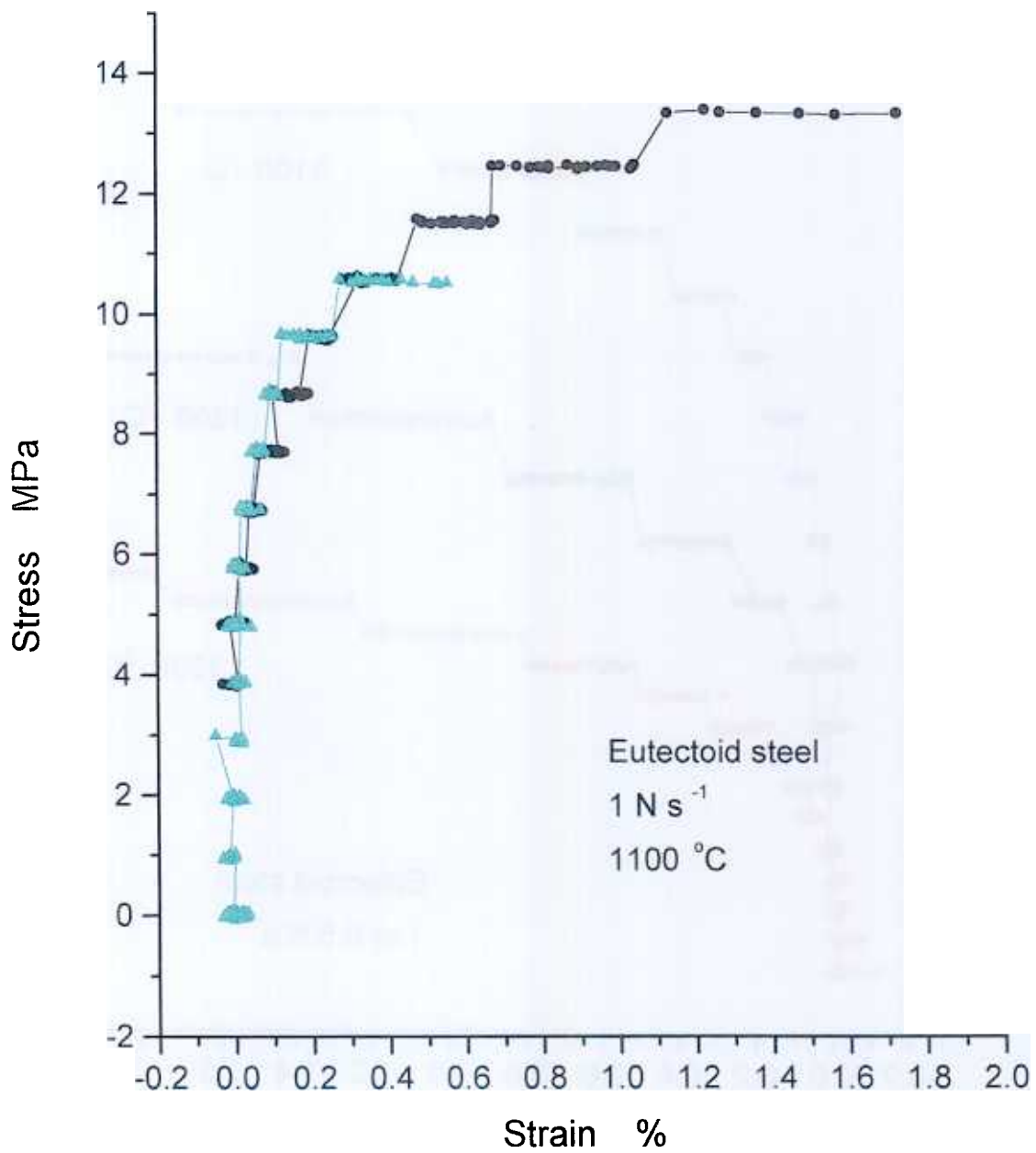


Fig 17 ETMT stress/strain curve at $1100 \text{ }^{\circ}\text{C}$ on the Eutectoid steel.

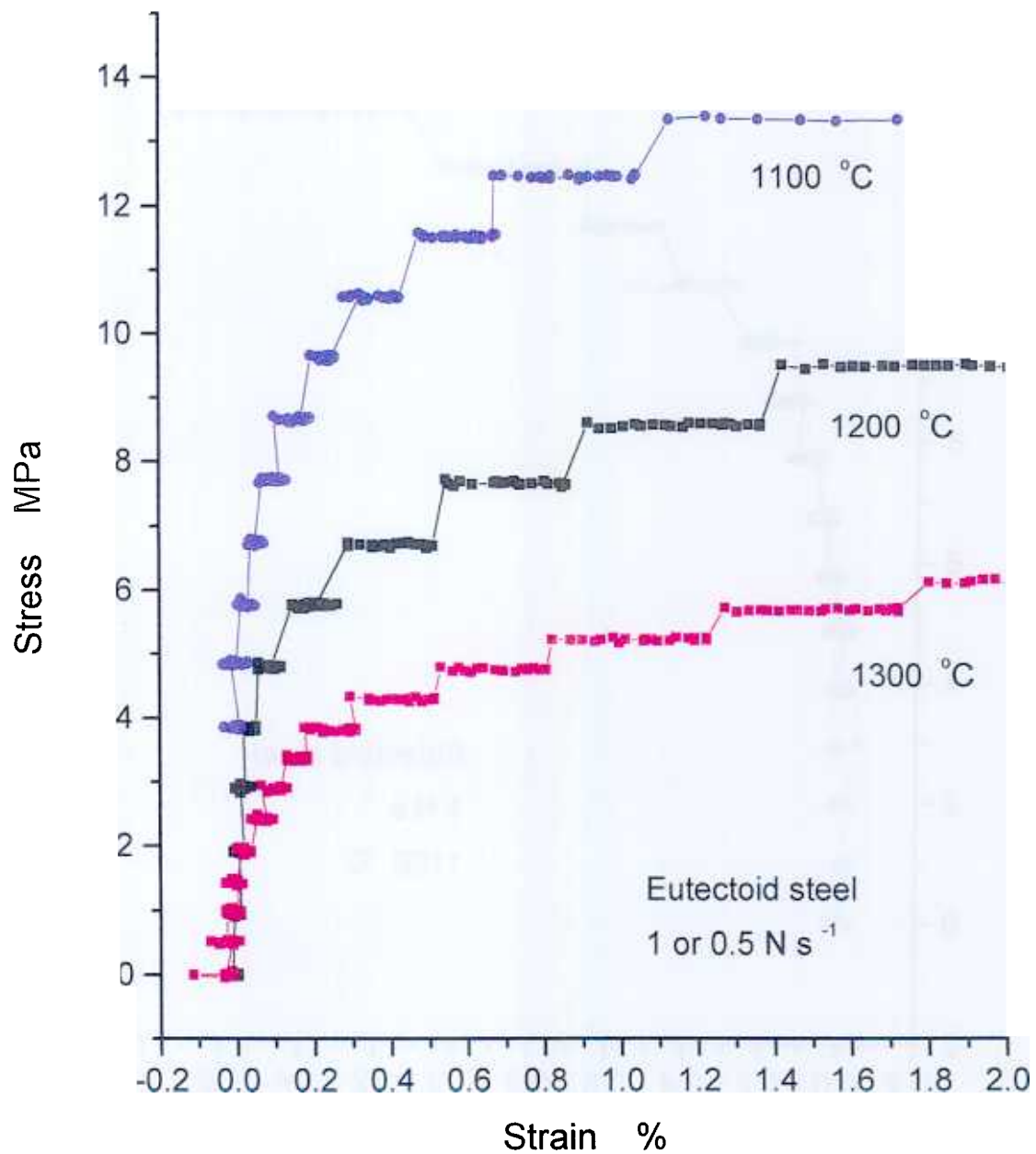


Fig 18 ETMT stress/strain curves on the Eutectoid steel at 1100-1300 °C.

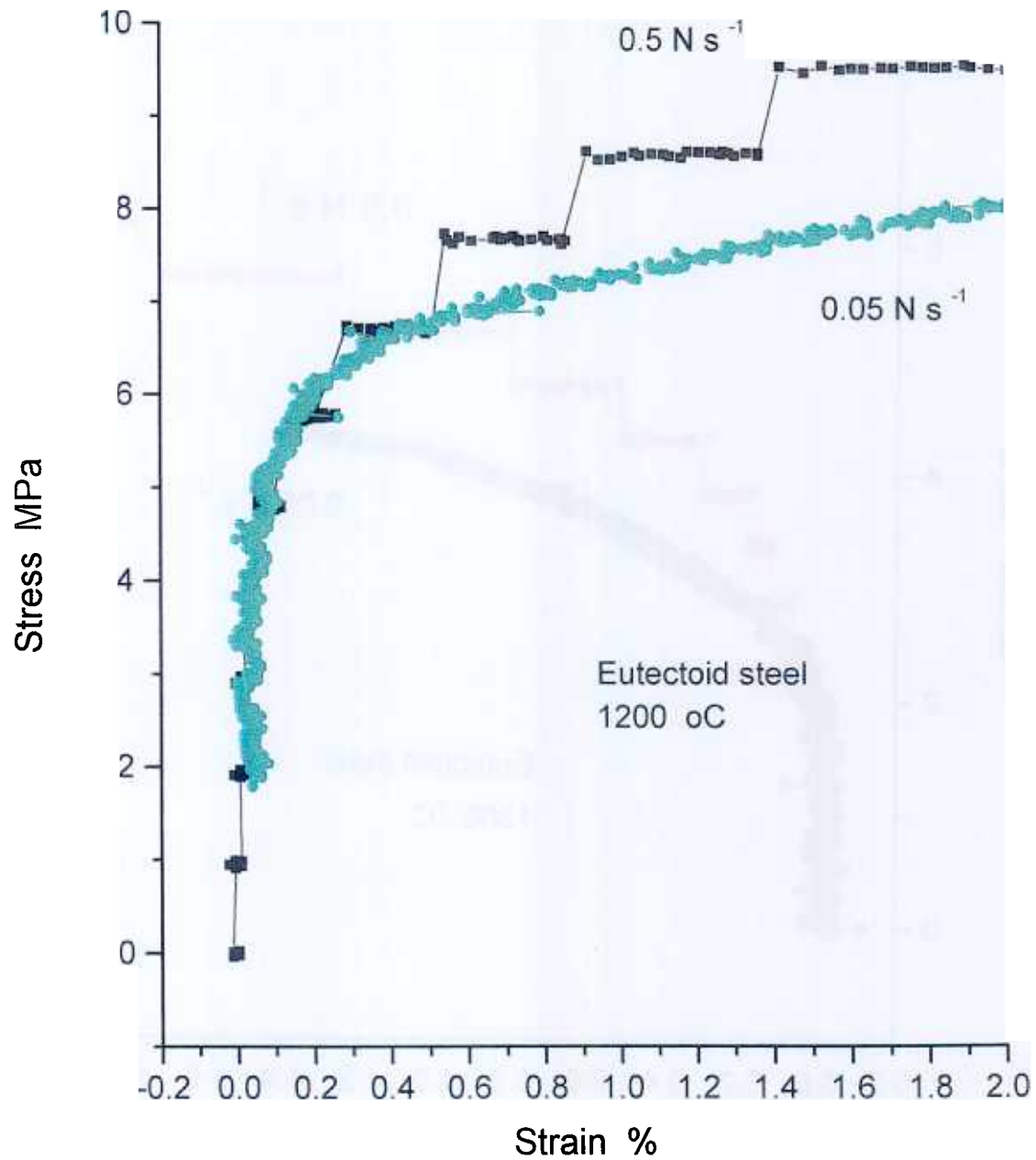


Fig 19 ETMT stress/strain curve at 1200 °C on Eutectoid steel showing strain rate effects.

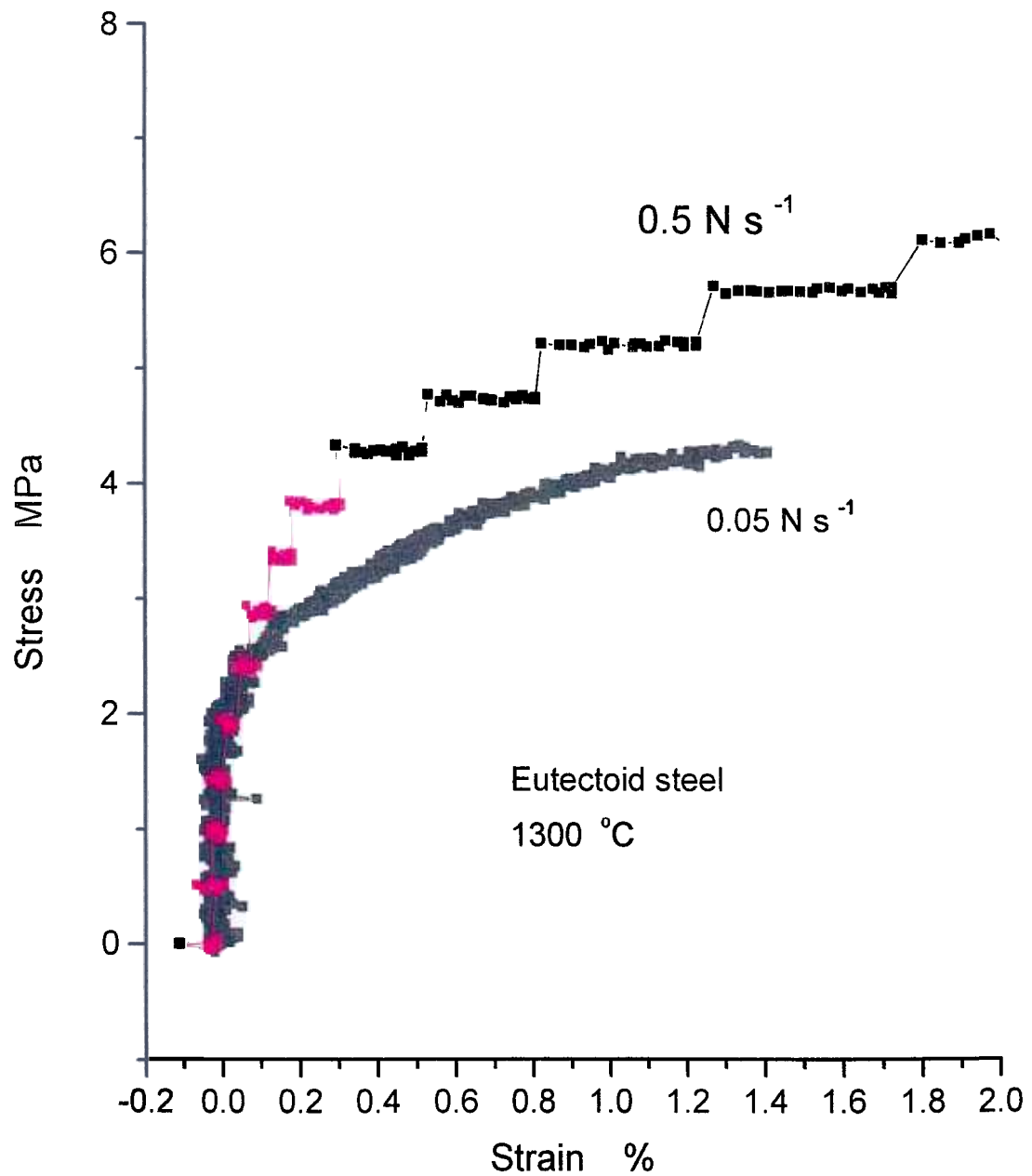


Fig 20 ETMT stress/strain curve at 1300 °C on Eutectoid steel showing strain rate effects.

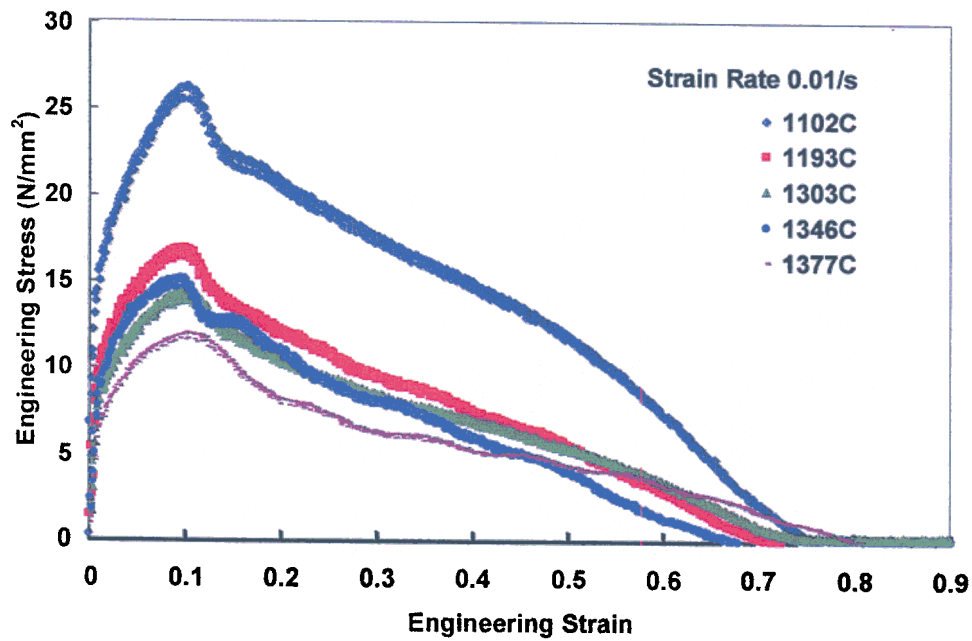


Fig 21 IHTR tests at 1100-1400 °C on the Eutectoid steel showing the effects of temperature and at 0.01 s⁻¹.

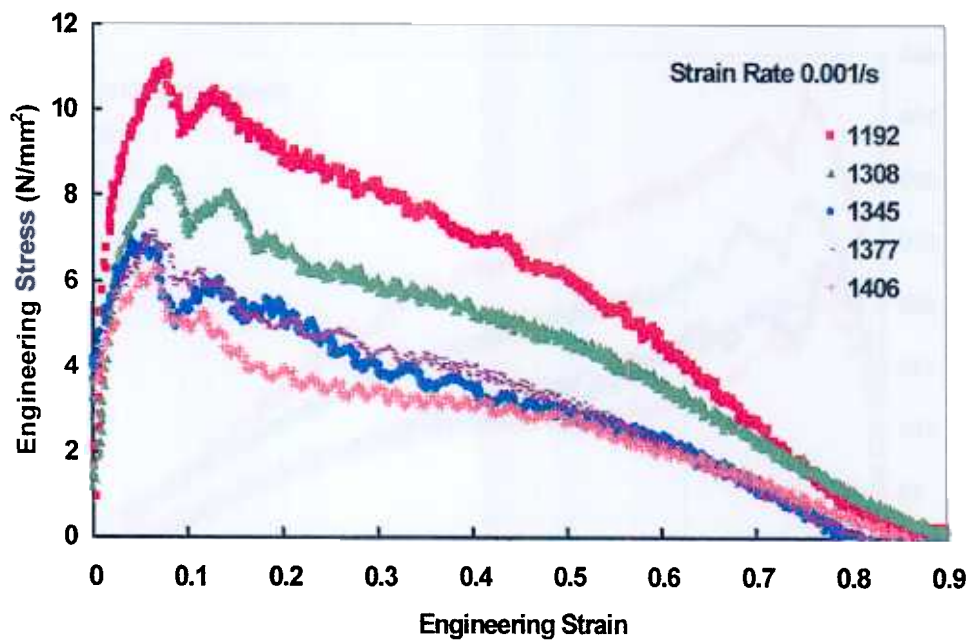


Fig 22 IHTR tests at 1100-1400 °C on the Eutectoid steel showing the effects of temperature and at 0.001 s⁻¹.

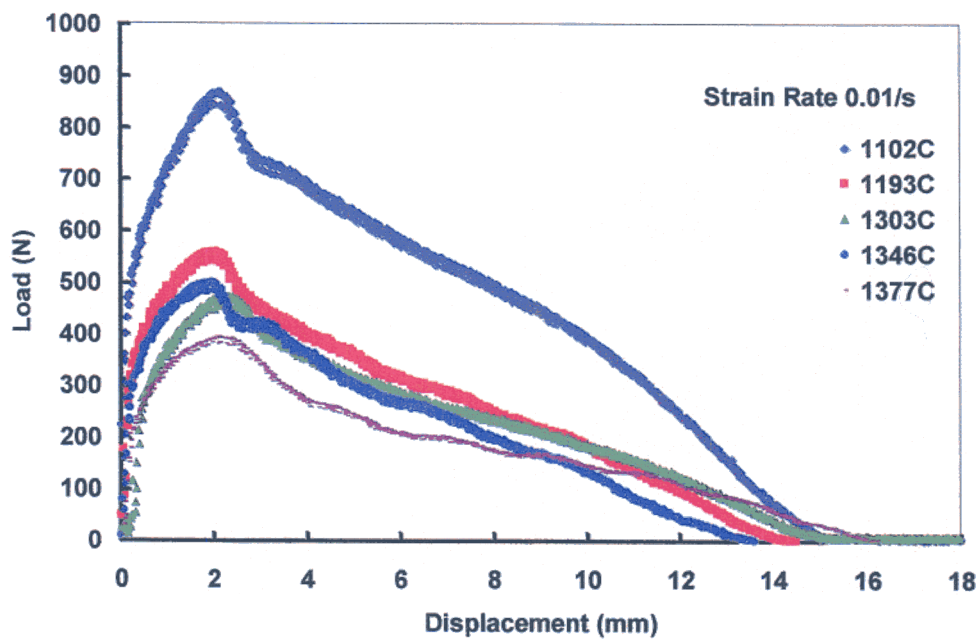


Fig 23 Load/displacement plots for IHTR tests on Eutectoid steel at 1100-1400 °C and 0.01 s⁻¹.

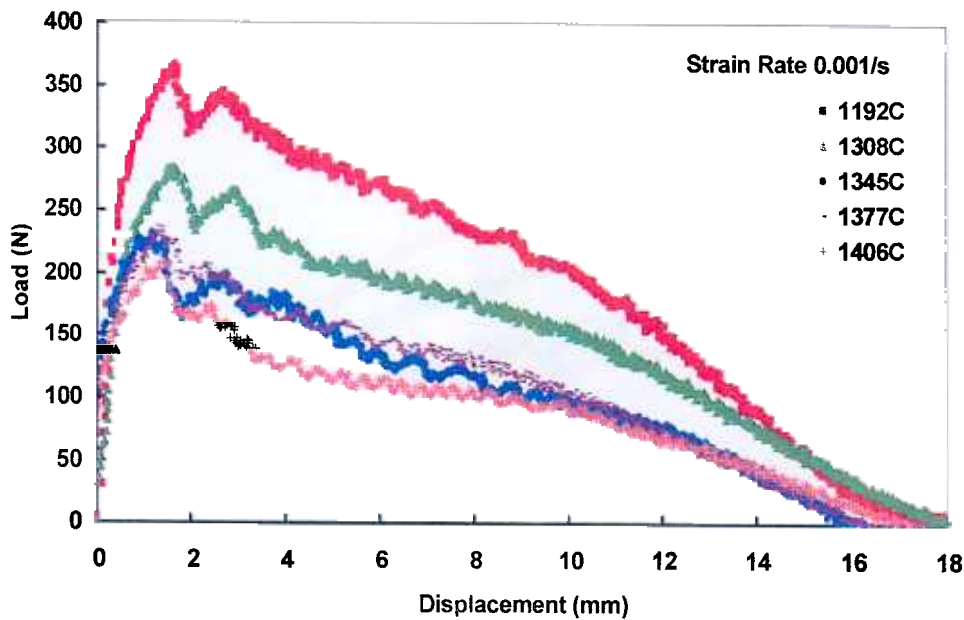


Fig 24 Load/displacement plots for IHTR tests on Eutectoid steel at 1100-1400 °C and 0.001 s⁻¹.

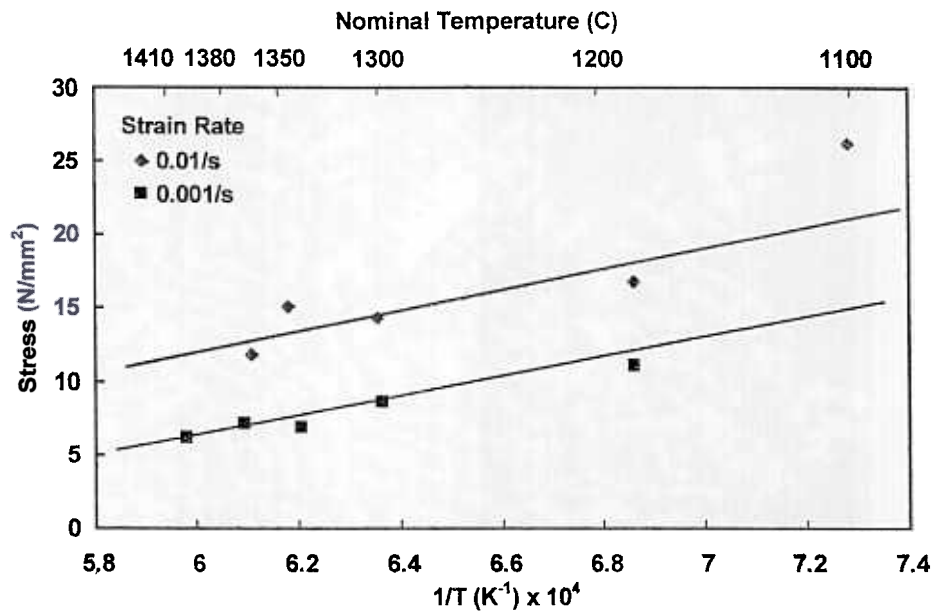
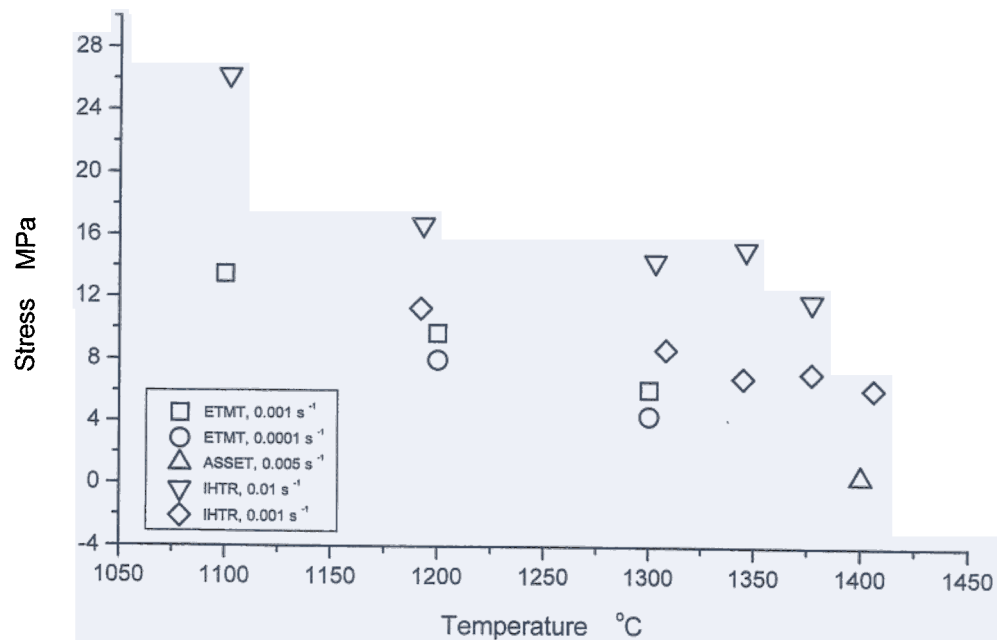


Fig. 8 Plot of Peak Stress vs $1/T$ for Eutectoid Steel

Plot of stress at the peak value against inverse temperature for the Eutectoid steel (IHTR data).



Comparison of Eutectoid steel strength data.

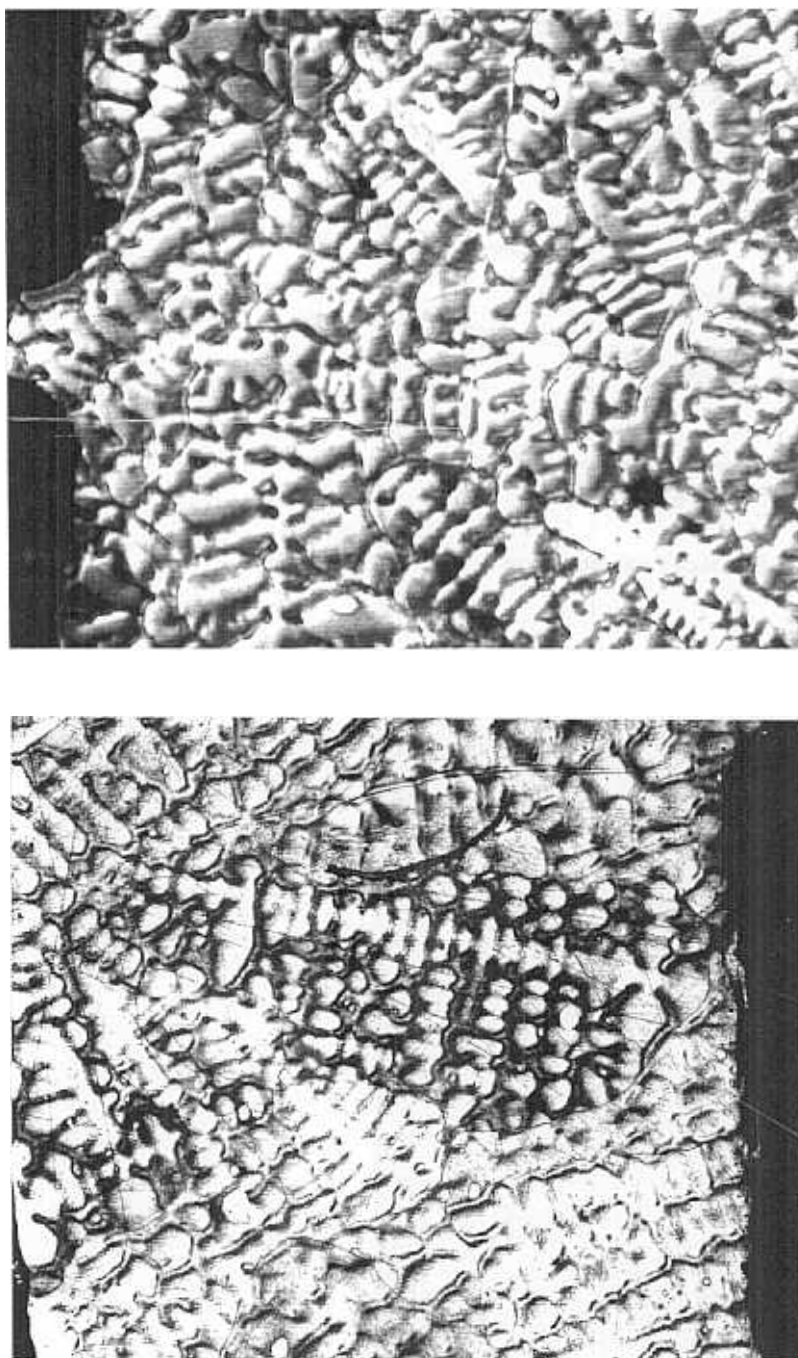


Fig 27 Dendritic microstructure of unmelted Cu-Ni testpiece (x5).

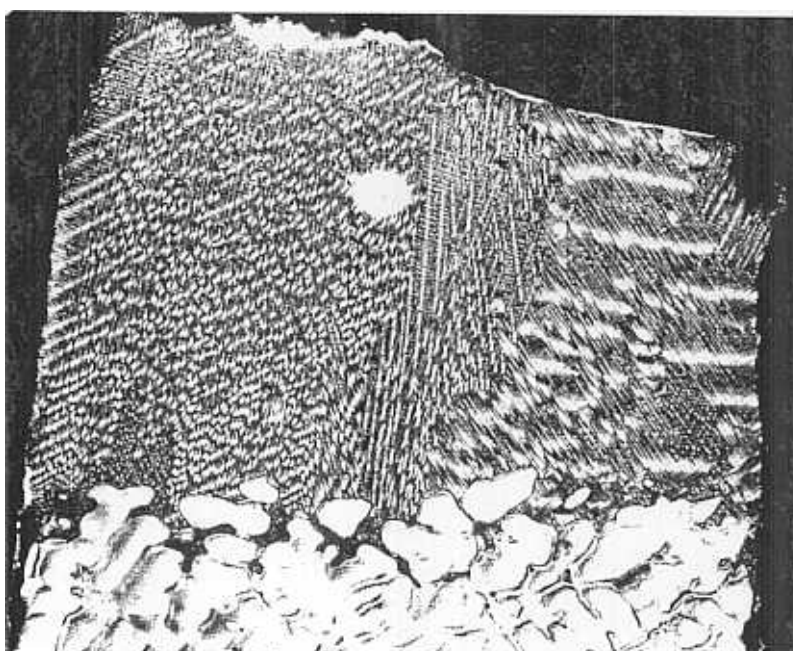
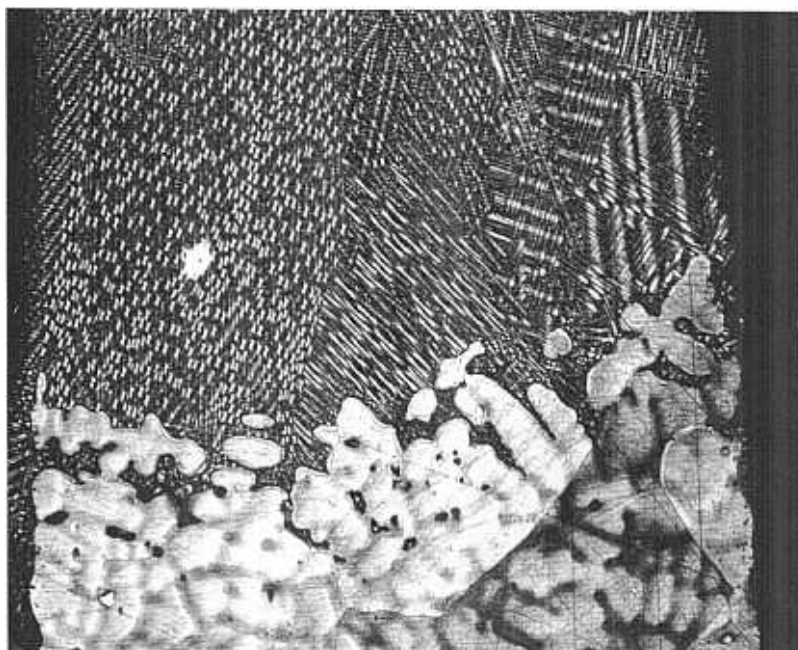


Fig 28 Refined dendrites in melted region of Cu-Ni testpiece (x5).

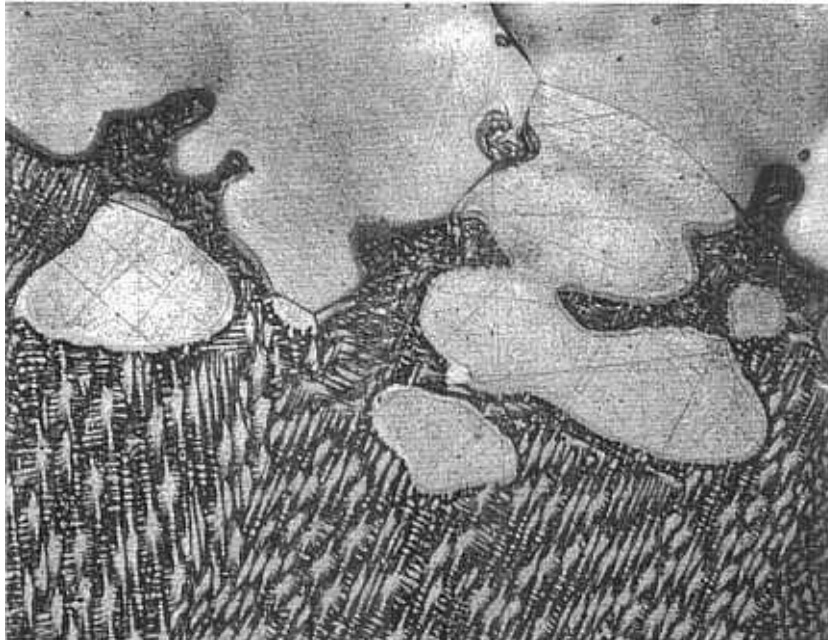


Fig 29 Structure at solid/liquid interface in Cu-Ni ETMT testpiece (x20).