

ORNL/M-5674

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**Proceedings of the IEA Working Group Meeting  
on Ferritic/Martensitic Steels**

JET Headquarters  
Culham, United Kingdom  
October 24-25, 1996

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Prepared by: R. L. Klueh

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**IEA WORKING GROUP - TASK ANNEX II**

Implementing Agreement  
for a program of research  
and development on fusion materials

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Implementing Agreement  
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and development on fusion materials

\*Research sponsored by the Office of Fusion Energy Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

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# IEA WORKSHOP AND WORKING GROUP MEETING ON FERRITIC STEELS

JET Headquarters, Culham, UK

24-25 October 1996

## Thursday 24 September 1995

08.20 Welcome and Introduction

### Work on IEA Heats and Other Heats

08.30 K. Ehrlich "Summary of Milestone 2 of EU Program

09.30 A. Kohyama, "Summary of the Irradiation Data on JLF-1"

10.00 K. Shiba, "Current status of Round-Robin tests in Japan: Unirradiated and Irradiated Results"

10.30 Break

10.45 D. S. Gelles, "Mixed-Mode Fracture Toughness in F82H"

11.15 A. Hishinuma, "Miscellaneous Results on F82H: Effects of Ti, N, Ta on Grain Size, Vacuum Properties, etc."

11.45 G. R. Odette, "An Integrated Approach to Assessing the Fracture Safe Margins of Fusion Reactor Structures"

12.30 M. Horsten, "New Information on Irradiated Modified 9Cr-1Mo Steel"

13.00 Lunch

14.00 Tour of JET

### Work on IEA Heats and Other Heats (Continued)

16.00 P. Spattig, "CRPP Data on Irradiation Programme in the PIREX Facility to 0.7 dpa/He: Effects on Radiation Hardening

- 16.30 A. Kohyama, "Recent Results on the Second Heat of JLF-1"
- 17.00 D. S. Gelles, "Recent Results on Impact Behavior of Irradiated Low Activation Steels"
- 17.30 R. L. Klueh, "Summary of Tensile and Charpy Properties of Irradiated ORNL Reduced-Activation Ferritic/Martensitic Steels"
- 18.00 Adjourn

Friday 25 October 1996

General Status of Fusion Materials Programs

- 09.00 W. Dietz: "Reorientation of the EU material programme"
- 09.45 A. Hishinuma, "Present Status of JAERI Program on LAM Development"
- 10.15 Break
- 10.30 A. Kohyama, "Present Status and the Future Plan on JLF-1 and the Japanese University Activities"
- 11.00 R. L. Klueh, "Status of U.S. Program"
- 11.30 Discussions and Recommendations for Future Cooperation
- 13.00 Adjourn

## **REPORT OF THE IEA WORKSHOP/WORKING GROUP MEETING**

### **ON FERRITIC/MARTENSITIC STEELS FOR FUSION**

**JET Headquarters, Culham, UK, 24-25 October 1996**

**R. L. Klueh**

#### **Executive Summary**

An International Energy Agency (IEA) Working Group on Ferritic/Martensitic Steels for Fusion Applications, consisting of researchers from Japan, the European Union, the United States, and Switzerland, met at the headquarters of the Joint European Torus (JET), Culham, United Kingdom, 24-25 October 1996. At the meeting, preliminary data generated on the large heats of steel purchased for the IEA program and on other heats of steels were presented and discussed. The second purpose of the meeting was to continue planning and coordinating the collaborative test program in progress on reduced-activation ferritic/martensitic steels. The next meeting is planned to be held in conjunction with the International Conference on Fusion Reactor Materials (ICFRM-8) in Sendai, Japan, 23-31 October 1997.

#### **Introduction**

The IEA Working Group on Ferritic/Martensitic Steels for Fusion under the auspices of the IEA Executive Committee for the Implementing Agreement on Fusion Materials met at JET Headquarters, Culham, UK, 24-25 October 1996. Researchers from Japan (3), the European Union (4), the United States (3), and Switzerland (1) participated. Russian Federation participation was invited, but no one from there attended the meeting. The objective of the Working Group is the establishment and coordination of an international collaborative test program to determine the feasibility of using ferritic/martensitic steels for fusion.

This was the sixth meeting of the Working Group, which was formed as a result of a workshop on ferritic/martensitic steels in Tokyo in October 1992. At the first meeting following the Tokyo workshop, the Working Group developed specifications for large heats of reduced-activation steels and outlined a collaborative research program. Two 5-ton heats of the IEA-modified F82H steel and two 1-ton heats of JLF-1 steel were produced, fabricated into plates, and distributed to the participants of the collaboration. Subsequent meetings were used to plan a test program and to coordinate the acquisition of the data needed to prove feasibility for the steels for fusion.

The Culham meeting was a follow up to the meeting at Baden, Switzerland, 19-20 September 1995, at which the first data on the new large heats were presented; the Culham meeting was

planned to expand on the review of data on the new heats, since the amount of data generated was expected to increase during the following year. The meeting was also set to review the status and future direction of the programs in Japan, Europe, and the United States in a continuing effort to coordinate the collaborative program. Working Group members who participated in the meeting and the meeting agenda are shown in the front of this report.

### Research and Development Activities

The first day of the Culham meeting consisted of a workshop on research results obtained on the large IEA heats of F82H and JLF-1 and research results on other ferritic/martensitic steels that have an impact on the objective of the Working Group. A range of properties have been examined for the large modified IEA heats of F82H and JLF-1 reduced-activation martensitic steels. These presentations are discussed in the first section below, followed by a section summarizing the work on other reduced-activation steels and non-reduced-activation steels. Copies of handouts of viewgraphs used for the presentations are attached to this report.

#### Experimental Work on IEA Heats

K. Ehrlich reviewed the studies of nine institutes throughout Europe that had combined to evaluate the homogeneity of chemical composition and mechanical properties of the plates from the IEA F82H heat. The plates are homogeneous, with little variation in grain size, hardness, and tensile properties through the thickness of the 7.5- and 15-mm plates produced from the large heat (the 15-mm plate is slightly stronger than the 7.5-mm plate). Heat treatment characteristics were determined for the steels in an effort to ascertain how optimum properties can be developed. Included in these studies were determinations of austenitization and tempering behavior, and a continuous cooling transformation diagram was determined. The properties of the IEA F82H steel was compared with other reduced-activation steels and the MANET conventional Cr-Mo steel. The IEA modified F82H heat had better Charpy properties than the MANET, but creep-rupture strength and the yield and tensile strength below 500°C of the IEA F82H were slightly less than those for the MANET. The ductility of the IEA F82H exceeded that for the MANET.

K. Shiba reported on the range of mechanical and physical properties planned and being carried out on the modified IEA F82H heat by JAERI. The planned mechanical properties test matrix includes tensile, creep, Charpy impact, fracture toughness ( $K_{Ic}$  and  $J_{Ic}$ ), fatigue, and hardness tests. These properties are being determined on the as-heat treated base metal and on weldments; tensile, Charpy impact, and fracture toughness data are also being determined on thermally aged base metal and weldments. The following physical properties are also to be determined: density, specific heat, thermal conductivity, thermal expansion, electrical conductivity, melting point, Young's modulus, Poisson's ratio, and magnetic hysteresis. As the handouts show, much of this characterization work has already been accomplished.

Other JAERI studies on the modified F82H were presented by A. Hishinuma. These included studies that showed the effect of tantalum and titanium on grain size and Charpy impact behavior. There were also studies on radiochemical analysis of impurities in the steel, vacuum properties (gas desorption), and corrosion behavior in high-temperature water. The corrosion tests were at 250 and 280°C and showed the effect of dissolved oxygen on the behavior. (A European paper on this subject presented by M. Maday/ENEA during the recent SOFT conference was transmitted to Dr. Shiba in accordance with an action item of the previous working group meeting.)

D. S. Gelles reported on mixed-mode fracture toughness of the modified IEA F82H and compared the results with previous results reported on in Baden on a small heat of F82H with a different heat treatment that he The previous results indicated that there was an effect of crack angle, and the toughness in the mixed mode is less than for either Mode I or Mode III. The IEA heat gave similar results for Mode I fracture toughness tests, but there were differences for mixed-mode I/III for the two materials. Tests are continuing to determine the cause of the differences.

P. Spatig reported on a program to study the effect of helium on the properties of the IEA F82H. To determine hardening effects, helium is to be injected into tensile specimens in either the PIREX facility in Switzerland or the dual-ion beam in Karlsruhe. For comparison, pre-injected specimens will be irradiated in the Studvik reactor in Sweden. Specimens irradiated in PIREX have been tested, and specimens are presently being irradiated in the Studvik reactor. Spatig also discussed studies under way to determine activation volumes for irradiated specimens using stress relaxation tests.

A. Kohyama reported on the extensive studies of the large heats of the JLF-1 steel. Just as is the case for the F82H, Monbusho researchers have conducted a range of microstructural and mechanical properties studies on the base metal and on TIG and EB weldments. Tensile, Charpy impact, and creep tests were conducted.

In general, the results of these tests indicate that the new IEA heats have properties comparable or better than those of the commercial steels they would replace.

#### Experimental Work on Other Heats of Steel

Several reports concerned information gathered on irradiated properties of other heats of reduced-activation and conventional steels, including F82H and JLF-1 heats other than the large IEA heats.

A. Kohyama summarized irradiation studies of Cr-W-V steels containing 2.25-12% Cr (including JLF-1 and F82H) irradiated in the FFTF/MOTA experiments. Tensile, Charpy, irradiation creep, and microstructural studies were conducted at 370-600°C and up to 60 dpa. One interesting

observation was that hardening as measured in a tensile test appears to go through a maximum with dose, which was also reflected in the shift in the transition temperature of the Charpy tests.

D. S. Gelles reported Charpy impact data for PNNL reduced-activation steels containing 2-12% Cr and different levels of manganese irradiated to 10 and 30 dpa in FFTF/MOTA at  $\approx 370^\circ\text{C}$ . There was no change in properties between 10 and 30 dpa. Manganese additions in the 9-12% Cr steels caused a change from cleavage to intergranular fracture. It was concluded that the 7-9Cr steels show the most promise.

K. Shiba reported on the tensile and fracture toughness properties of a heat of F82H irradiated in HFIR over the range  $200\text{-}500^\circ\text{C}$ . Experiments to assess helium effects using boron doping were also reported. Different levels of  $^{10}\text{B}$  were added to F82H, and the steels were irradiated in the JMTR to 0.05-0.6 dpa at 400 and  $550^\circ\text{C}$ . No effect of helium was observed on tensile properties, but there appeared to be a slight effect on the Charpy behavior.

A. Kohyama reported on work by Kimura, Morimura, and Matsui of Tohoku University who studied the effect of helium implantation on the DBTT of a 9Cr-2WVTaB steel. TEM disks were implanted with 120 appm He in a cyclotron at 36 MeV at  $\approx 220^\circ\text{C}$  to 0.048 dpa. A small punch test was used to estimate an increase in yield stress of  $\approx 104$  MPa and a shift in the DBTT of  $\approx 50^\circ\text{C}$ . The changes were attributed to the possibility of enhanced hardening by helium.

M. G. Horsten reported on irradiation studies of tensile and fracture toughness measurements of modified 9Cr-1Mo and Sandvik HT9 irradiated at  $\approx 80$  and  $300^\circ\text{C}$  in the High Flux Reactor (HFR) at Petten. Tensile tests from room temperature to  $600^\circ\text{C}$  showed that the high-temperature damage ( $300^\circ\text{C}$  irradiation) was more stable than low-temperature damage ( $80^\circ\text{C}$  irradiation). The toughness of the modified 9Cr-1Mo was superior to that of HT9.

R. L. Klueh reported on the tensile and Charpy impact properties of the ORNL reduced-activation steels with 2.25-12% Cr irradiated in FFTF/MOTA to 27-29 dpa. The 9Cr-2WVTa steel continued to show excellent impact properties. The shift in transition temperature was only  $32^\circ\text{C}$  after  $\approx 28$  dpa, although there appeared to be a slight increase in the shift with increasing fluence. However, even after the  $32^\circ\text{C}$  shift, the DBTT after irradiation was lower than the DBTT of modified 9Cr-1MoVNb or Sandvik HT9 before irradiation (these steels showed shifts of  $\approx 45$  and  $160^\circ\text{C}$ , respectively, for these irradiation conditions).

K. Ehrlich presented the latest Charpy impact data from a continuing irradiation experiment in HFR, in which the conventional steels MANET I and II and the reduced-activation steels OPTIFER Ia and II, F82H, and ORNL 9Cr-2WVTa are being irradiated. Data obtained after irradiation to 2.5 dpa at  $300^\circ\text{C}$  were reported; previously, data were reported to 0.8 dpa. The reduced-activation steels again performed better than the MANET steels, and the 9Cr-2WVTa steel again showed the smallest change in Charpy properties.



### Fracture Assessment

An integrated approach to assessing fracture-safe margins of fusion power plant structures was presented by G. R. Odette. The types of data on irradiated and unirradiated specimens required to apply the methodology were outlined, and examples of the application of the technique were described. This approach has been formalized in a paper that was the subject of an action item from the Baden meeting.

### Collaboration: Planning and Status

On the second day of the meeting, the status and future direction of the programs in Europe, Japan, and the United States were reviewed by W. Dietz, A. Hishinuma, A. Kohyama, and R. L. Klueh, in a continuing effort to coordinate the collaborative program. Copies of the viewgraphs used for these presentations are attached.

A major question concerning the use of ferritic/martensitic steels for fusion has concerned the interaction of a ferromagnetic structural material with the magnetic fields of a fusion power plant and how the ferromagnetic material will affect plasma control. Up until now, this question has been addressed by design studies. In his discussion of the JAERI program, A. Hishinuma presented information on JAERI plans for an experimental study of this problem. JAERI will produce a simulated ferritic steel vacuum vessel by covering the inner surface of the vacuum vessel of their JFT-2M with F82H. Using this vessel, they hope to measure the effects of the ferromagnetic material on the operation of the machine. They also have plans for simulating a ferritic steel blanket module. Diagrams of the JFT-2M vessel with the proposed F82H modifications are shown in the attachment.

During the discussion of the proposed JAERI program, it was recognized by the Working Group that a timely assessment of the effect of a ferritic steel blanket structure on the ability to control a plasma is of utmost importance in verifying the feasibility of using ferritic steels for fusion. The program will also provide invaluable information on the development of large-scale fabrication technology of martensitic steels, because the project will require studies on the production of large heats of steel and the welding and joining of large structures. The Working Group and the fusion community will look forward to seeing the results of the JAERI study, because of the important contribution it will make toward our understanding of the martensitic steels for fusion applications.

Areas requiring coordination by the Working Group in the future were discussed. These include work on corrosion and hydrogen embrittlement and on creep-fatigue studies being carried out in the different programs. Action items were recommended on these subjects. In the near future, the Japanese and the European Union programs intend to purchase new large heats for component studies, and they intend to coordinate the specification effort for these heats. The need for a compilation of the data being obtained on the new heats of steels was considered. A

data base for F82H already exists at JAERI, and an action item was proposed for investigating the use of this data base.

#### Next Meeting

The next meeting was tentatively set for 3-4 November 1997 in Japan in conjunction with ICFRM-8 (23-31 October 1997). The Japanese delegation suggested that the meeting be held near the site of a Japanese steel plant, so that a tour of the facility could be made, thus providing an opportunity for the group to become informed on the technology of steelmaking to be used to produce ferritic/ martensitic steels for fusion applications. A. Hishinuma will make arrangements for the site of the next meeting.

#### Action Items

The following summarizes the action items developed as a result of the meeting:

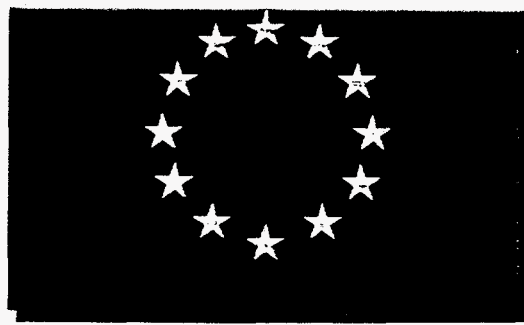
1. K. Ehrlich is presently compiling a report on the results of the European Union tests to study the homogeneity of the large IEA heat of modified F82H. To broaden the scope of the report, Ehrlich is willing to include any Japanese and U.S. data on this subject in the appendix of the report. The data must reach Ehrlich in the next month to be included.
2. Corrosion and hydrogen embrittlement test programs are being conducted in Japan and the European Union. In accordance with an action item from the previous meeting, Dietz and Shiba/ Hishinuma exchanged program plans to ensure maximum information is derived from the tests. The corrosion programs are evolving and expanding, and the coordination of the two programs by these individuals will continue.
3. The European laboratories successfully conducted round-robin tests of miniature Charpy specimens during the last year. Three U.S. organizations agreed to participate, and specimens were distributed to them by Karlsruhe. These organizations are urged to test the specimens to expand the scope of the test comparisons.
4. As the present meeting demonstrated, considerable amounts of physical and mechanical property data have been collected and will be collected in the future on the large heats of modified F82H and JLF-1 that are part of the IEA collaboration. For these data to be of maximum use to the materials community and to designers, the data must be compiled and easily accessible. The Japanese (JAERI) have established a data base for this purpose, and Shiba will circulate to members of the Working Group information on the data base, including how data can be input, used, and exchanged by e-mail.
5. The European Union and Japanese fusion materials programs have plans to conduct component testing on F82H. To carry out such a program, new large heats of steel will need to be purchased. To assure that the quality of these new heats is maximized by taking into account

the information derived from the IEA program and other work, the purchasers of the new heats will consult within the Working Group in determining the specifications for the heats. The European Union will draft a specification by the end of January 1997.

6. The European Union has generated a test matrix to study the thermal fatigue of F82H. The matrix will be circulated to members of the Working Group for comment and an indication of interest in participation by other parties so that a detailed study of fatigue, including creep-fatigue interaction, can be completed as soon as possible. Tavassoli will comment on the program based on his knowledge from recent studies on this topic in the framework of the FBR program.

7. The effect of irradiation on the fracture behavior of ferritic/martensitic steels is the chief concern for the application of the steels to fusion. Odette has proposed a methodology to assess the fracture characteristics to allow for the prediction of the changes to be expected due to irradiation. For the system to be evaluated, it is necessary to obtain the appropriate data on unirradiated and irradiated material. To aid in that evaluation, it is proposed that the irradiation programs in progress be examined for ways to develop the data required for such an evaluation. Odette is already in contact with CRPP, and test plans for that program may be of interest for all.

# VIEWGRAPHS OF PRESENTATIONS



# **IEA-Working Group Meeting on Ferritic-Martensitic Steels**

**JET-Headquarters, Culham UK  
24-25 Oct 1996**

**Summary of the Milestone 2 - Meeting  
of the European Programme**

**Compiled by Karl Ehrlich  
Contributions by CEA-Saclay, CIEMAT-Madrid,  
ECN-Petten, ENEA-Casaccia, EPFL-Villigen,  
FZK-Karlsruhe, HMI-Berlin, RNL-Risø, VTT Espoo**

**References:**

- R. Lindau (Ed.): Homogeneity Tests of European Laboratories on Alloy F82H-mod.FZK-Report 5814, November 1996  
E. Daum, K. Ehrlich, M. Schirra (Eds.): Milestone 2 - Meeting on F/M - Steels in the European Programme, FZK-Report 5848, November 1996

# **New Results of Homogeneity Tests on F82H mod. (Heat 9471)**

**compiled by R. Lindau,  
with contributions of  
CEA, CIEMAT, ECN, ENEA, EPFL-PSI, FZK, NFR, VTT**

Karlsruhe, Sept. 9-10, 1996  
Culham, Oct. 24-25, 1996



## **New Results from the Homogeneity Tests on F82H mod. in Europe**

### **Chemical Composition**

- New analyses by Adelhelm, Daum (FZK) with different methods show that the manufacturer's analysis concerning Nb and Mo is correct.

### **Tensile Tests**

- No difference in tensile properties within one plate.
- No texture in tensile properties.
- 15 mm plate is slightly higher in strength and slightly lower in ductility than 7.5 mm plate.
- Tensile results of different labs show good agreement.
- Deviations in total elongation are due to different specimen geometry.
- Strength is below 500 °C lower, above 500 °C equal MANET II.
- Ductility is comparable to MANET II.

### **Impact Bending Tests**

- Round robin tests reveal rather little scatter in upper shelf energy (USE: 9.3-10.4 J)

and

- large scatter in DBTT, (-45 °C - 80 °C).

# Chemical Composition of F82H mod. (Heat 9741)

	C	Mn	Cr	V	W	Nb	Mo
JAERI	0.09	0.16	7.64 - 7.71	0.16	1.94 - 1.97	0.0001	0.003
EU	0.086 - 0.107	0.16 - 0.21	7.40 - 8.36	0.14 - 0.17	1.88 - 2.10	< 0.001 - 0.010	< 0.002 - < 0.01

	Ni	Cu	Al	Zr	Si	Ti	Co
JAERI	0.02	0.01	0.003	1)	0.11	0.006 - 0.008	0.005
EU	0.002 - 0.022	0.003 - 0.006	< 0.001 - 0.004	0.01	0.10 - 0.23	< 0.001 - 0.007	0.0037 - 0.009

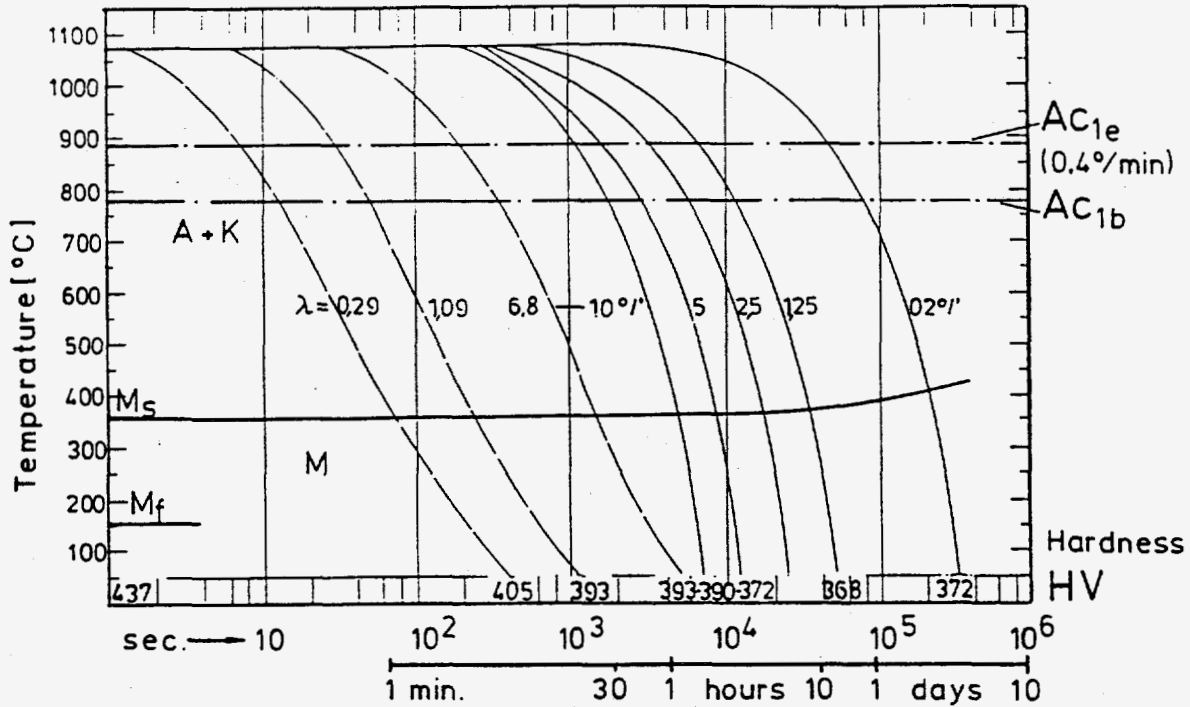
1) under evaluating

## ► New analyses

	JAERI	FZK Adelhelm	FZK Daum
Nb (wt-ppm)	1	< 8	2.5 ± 1
Mo (wt-ppm)	30	18 ± 1	46 ± 17



Material: MANET-II Heat-Nº: 50806  
 Austenitizing: 1075°C 15 min. Grain-size(ASTM): 9-10



Material: F82H - mod. Heat-Nº: 9741  
 Austenitizing: 1040°C 15 min. Grain-size(ASTM): 7-8, +6

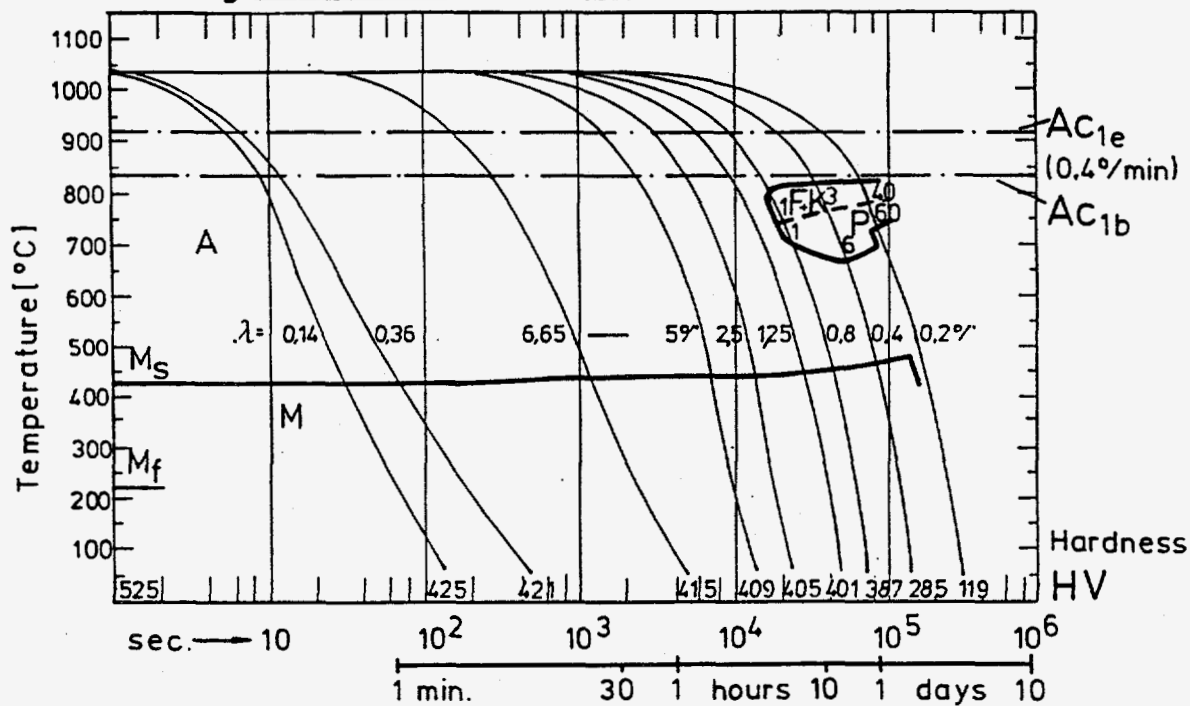
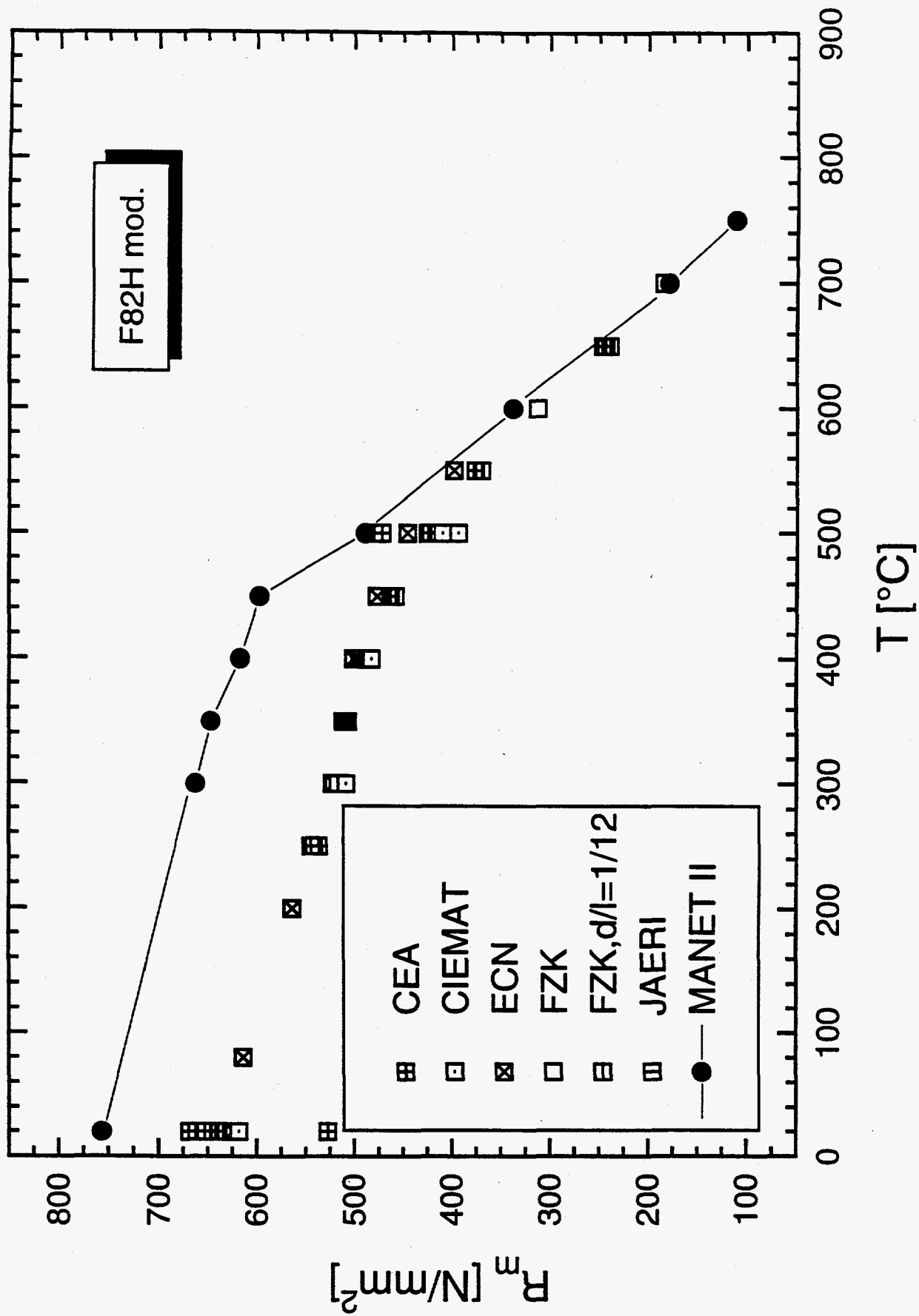


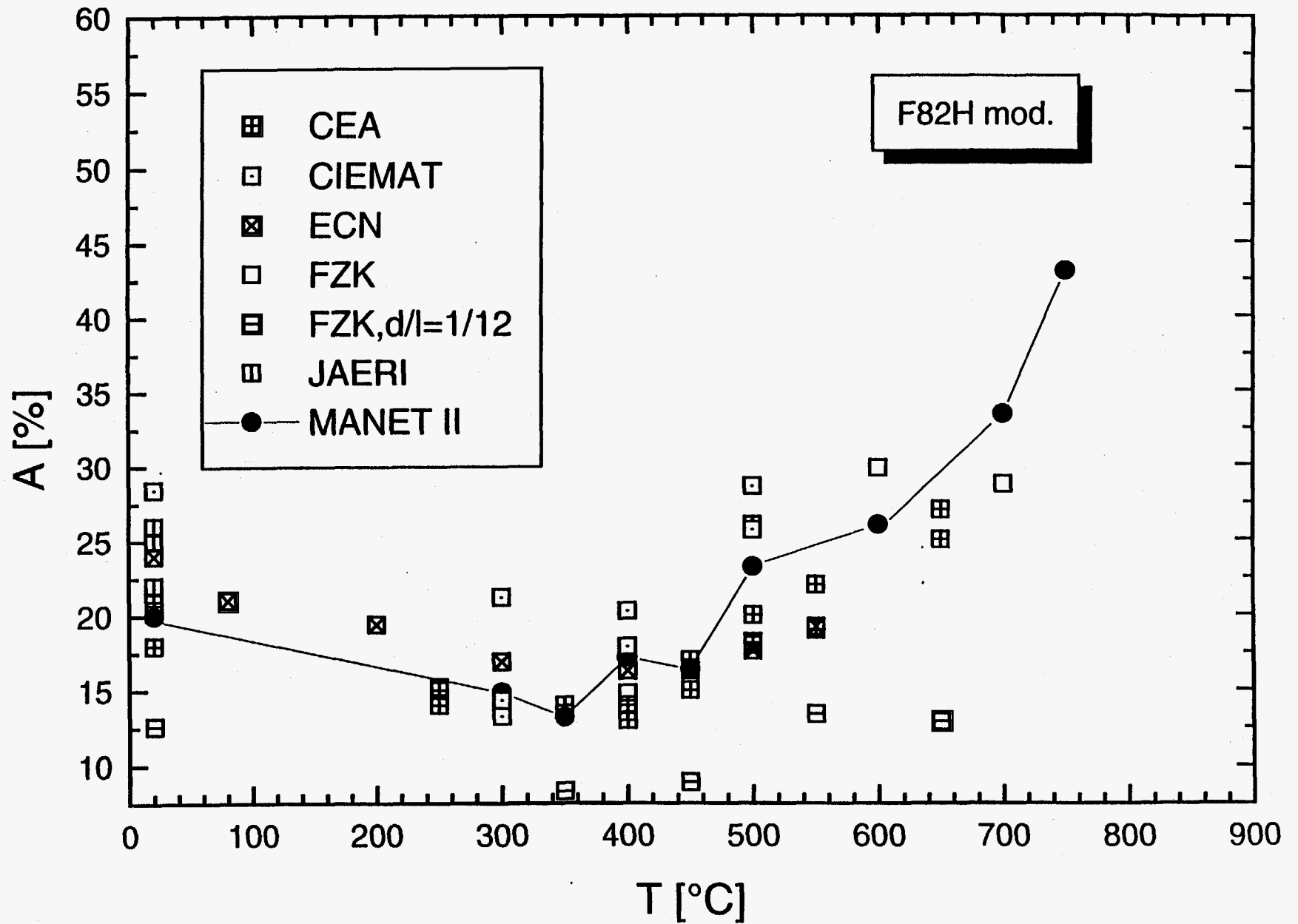
Fig: TTT-diagram for continuous cooling

# Ultimate Tensile Strength



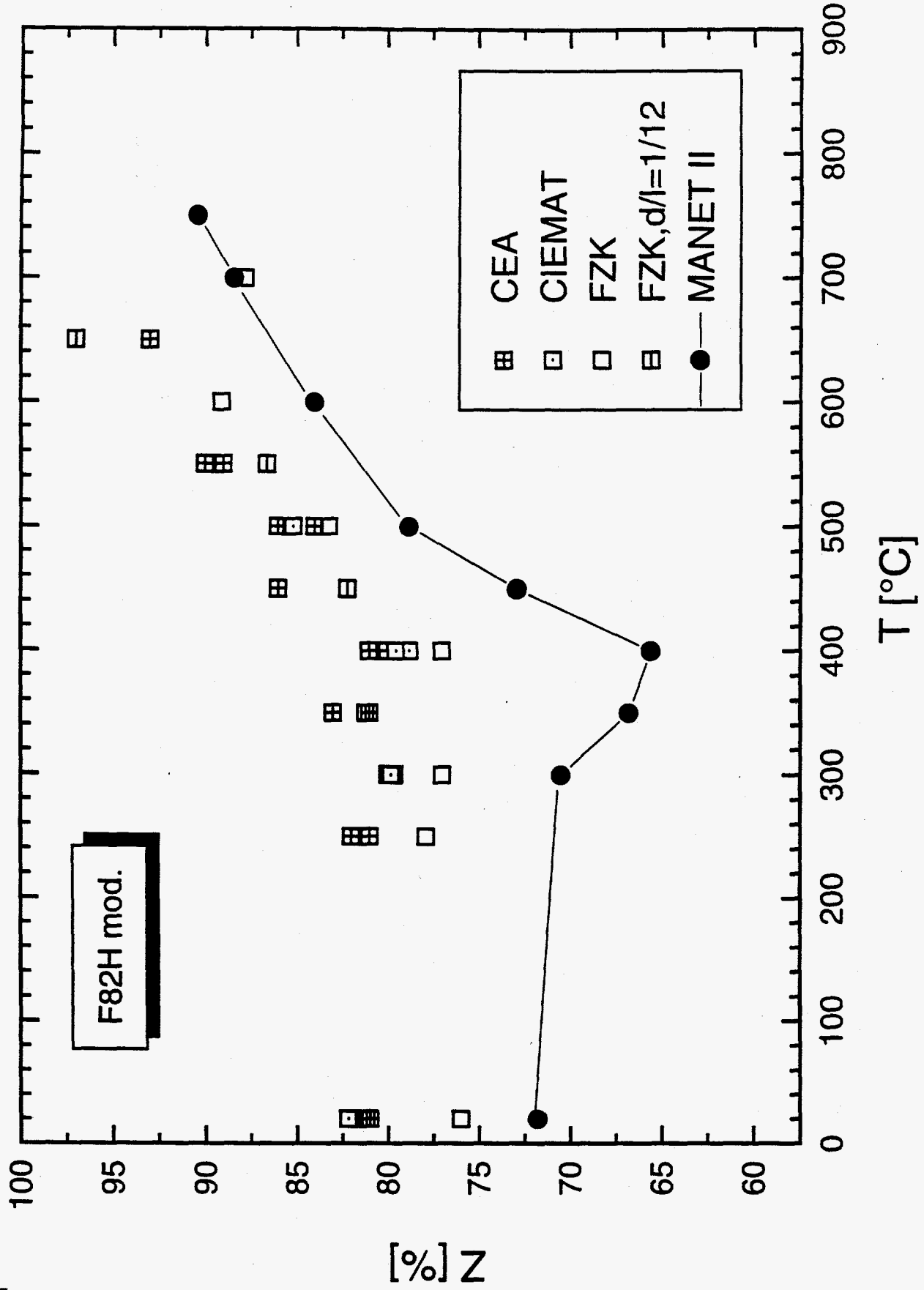


# Total Elongation





# Area Reduction

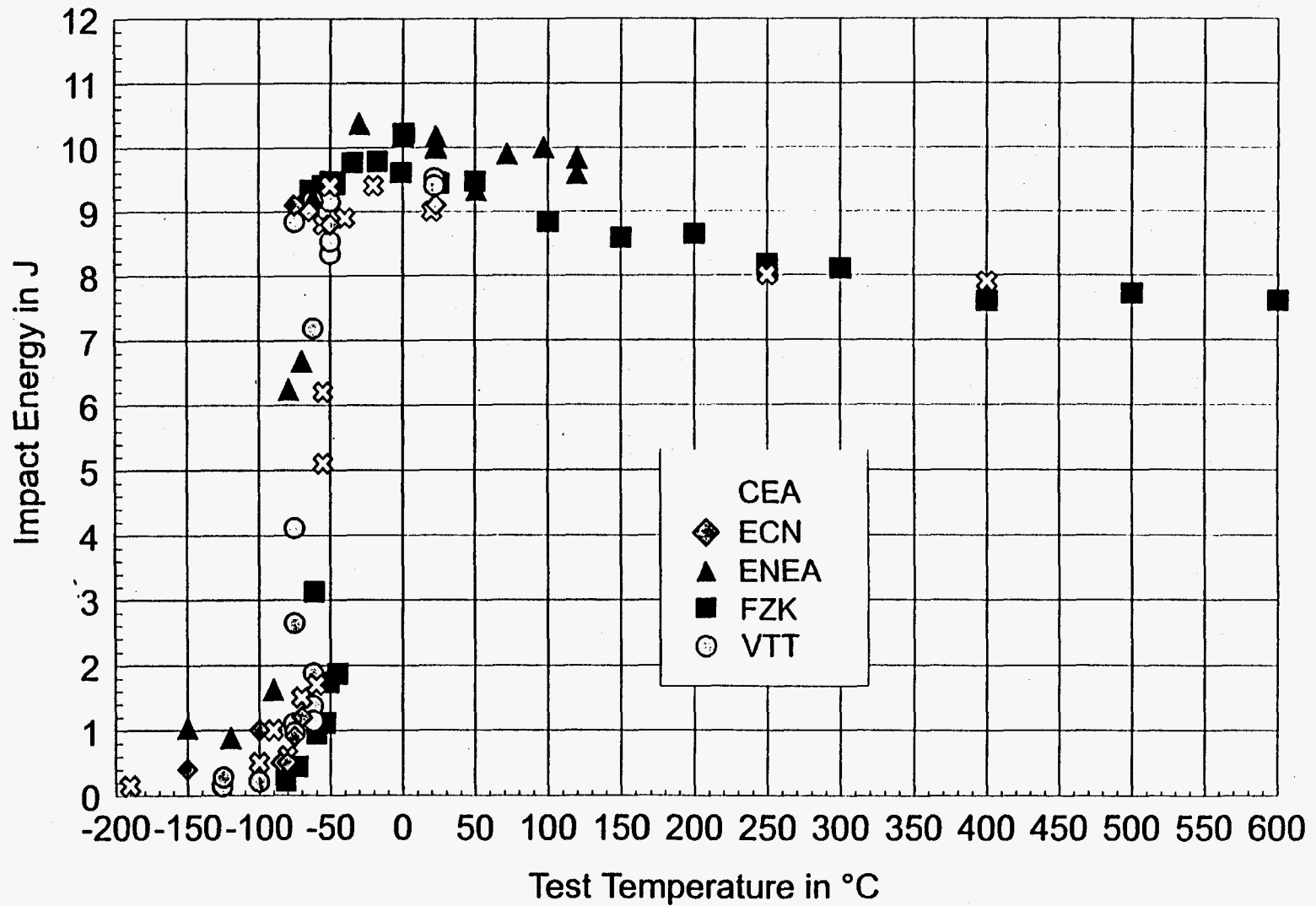


# Forschungszentrum Karlsruhe

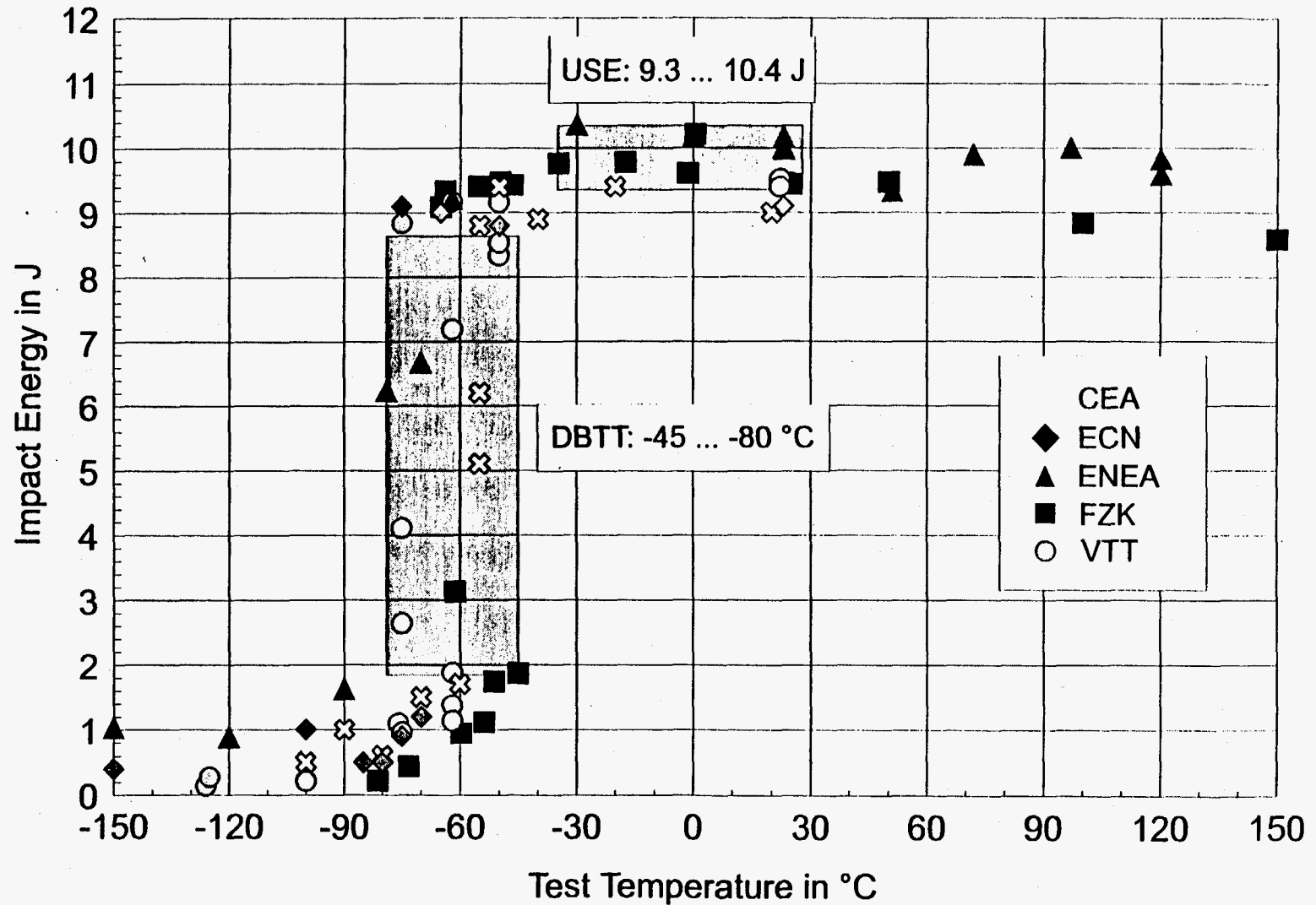
Technik und Umwelt

Institut für Materialforschung II

Leiter: Prof. Dr. D. Munz



Forschungszentrum Karlsruhe  
 Technik und Umwelt  
 Institut für Materialforschung II  
 Leiter: Prof. Dr. D. Munz





# European LA Ferritic-Martensitic Experimental Alloys

Association	Alloy-Name	C	Si	Mn	Cr	V	W	N	Ta	Ti	other
FZK/IMF	Optifer-Ia 1)	0.10	0.06	0.50	9.3	0.26	0.96	0.0155	0.066	-	Ge
	Optifer-II 2)	0.125	0.04	0.49	9.5	0.28	0.006	0.0159	0.018	-	1.2
EPFL/PSI	OPTIMAX-A 3)	0.094	0.002	0.56	9.24	0.24	2.00	0.0007	0.08		
ENEA	BATMAN-IC 4)	0.125	0.027	0.52	8.32	0.20	1.44	0.0072	-	0.009	
	BATMAN-IIC 5)	0.135	0.0275	0.54	7.50	0.235	1.425	0.0037	-	0.009	
CEA	LA 12 LC 6)	0.089	0.03	1.13	8.92	0.30	0.73	0.0350	0.10		
	LA 12 Ta LC 7)	0.090	0.03	1.13	8.80	0.30	0.73	0.0190	0.09		
	JLF-1 8)	0.106	0.05	0.52	8.70	0.18	1.91	0.0280	0.08		
IEA-Alloy	F82H mod. 9)	0.09	0.11	0.16	7.66	0.16	2.00	0.005	0.02	0.01	

1) = 1075 °C 30' + 750 °C 2h

2) = 950 °C 2h + 750 °C 2h

3) = 1050 °C 30' + 750 °C 2h

4), 5) = 1020 °C 1h/air + 1020 °C 1h/air + 730 °C 1h/air

6), 7) = 1100 °C 1h + 675 °C 2h or 750 °C 2h

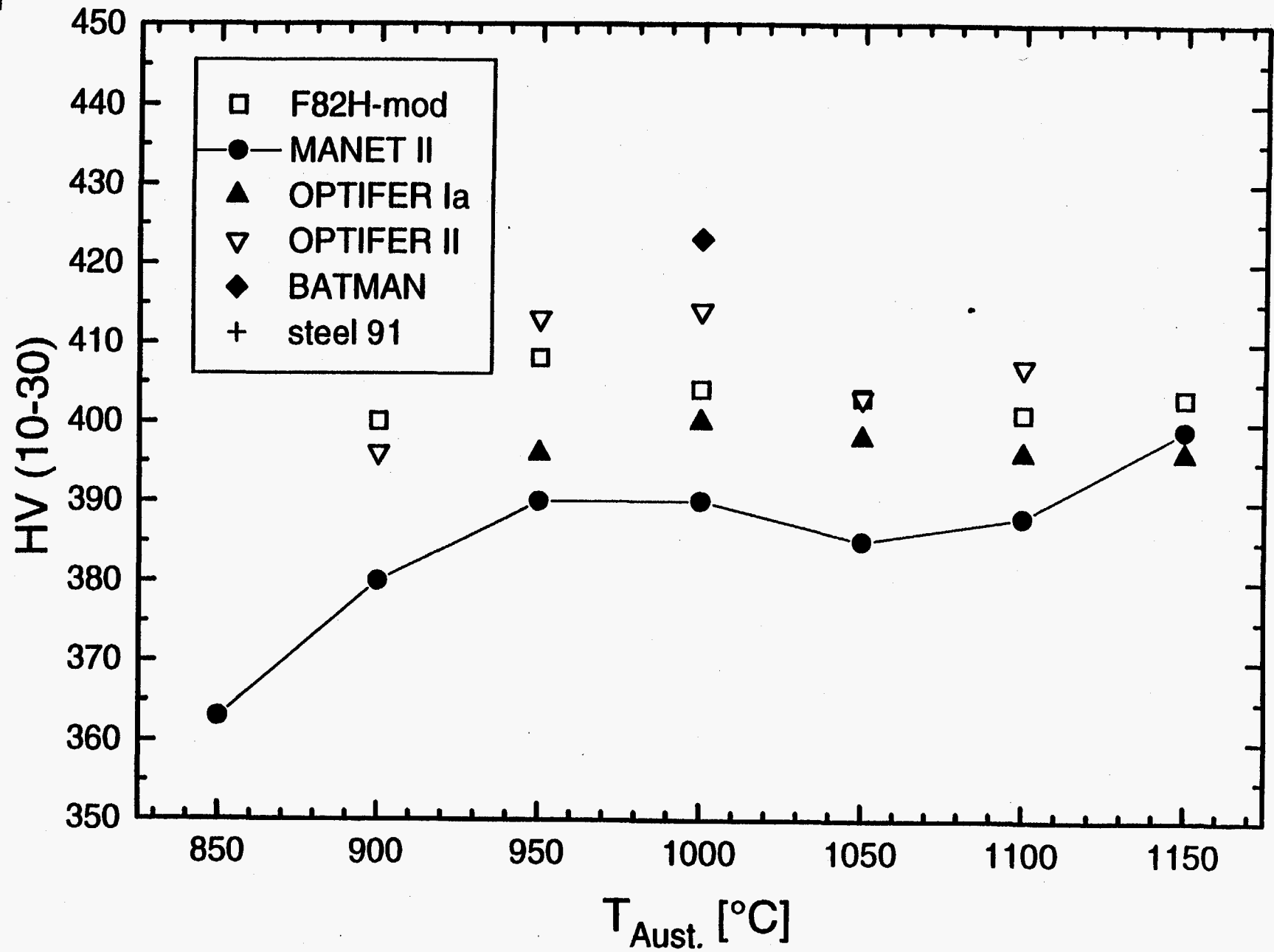
8) = 1050 °C 1h + 780 °C 1h

9) = 1040 °C 40' + 750 °C 1h

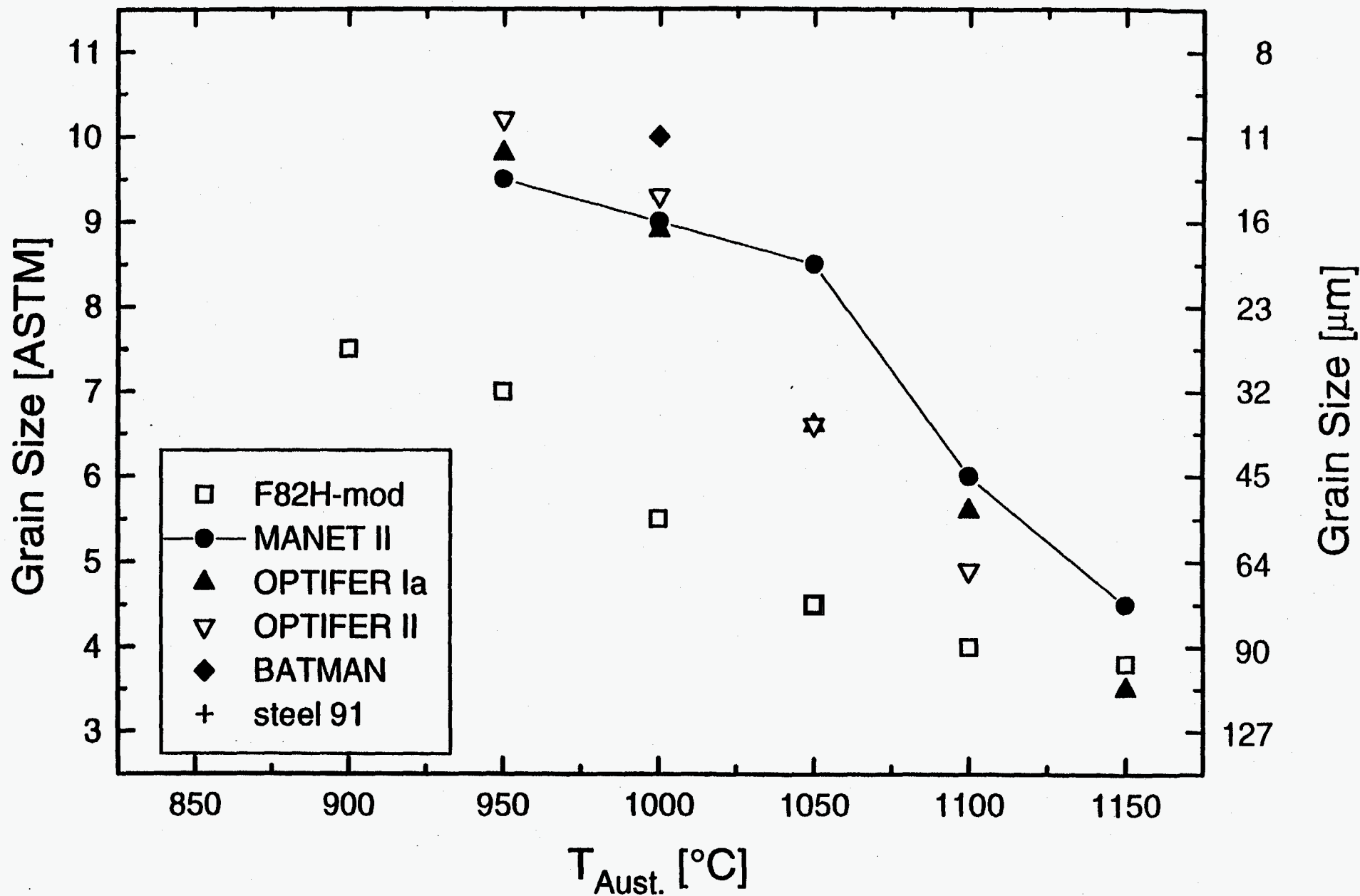
Transformation points

	Ac <sub>1b</sub>	Ac <sub>1e</sub>	M <sub>s</sub>	M <sub>f</sub> (°C)	pearlite transformation start (cont.cooling)
F82H-mod heat9741	835	915	425	220	1 °/min
MANET II 6 heats	775-780	890-900	340-357	155-161	< 0,2
OPTIFER Ia heat 664	820	900	418	222	4
OPTIFER II heat 668	825	920	395	172	2,5
BATMAN 2 heats	837-843	911-913	430-440	215-230	0,7
Steel 91 3 heats	810-820	870-885	385-400	100-120	3-4
OPTIMAX					
LA13Ta	821	925	325		1,25

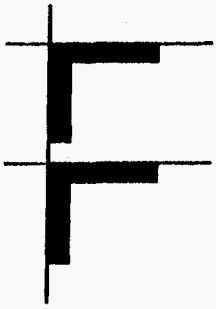
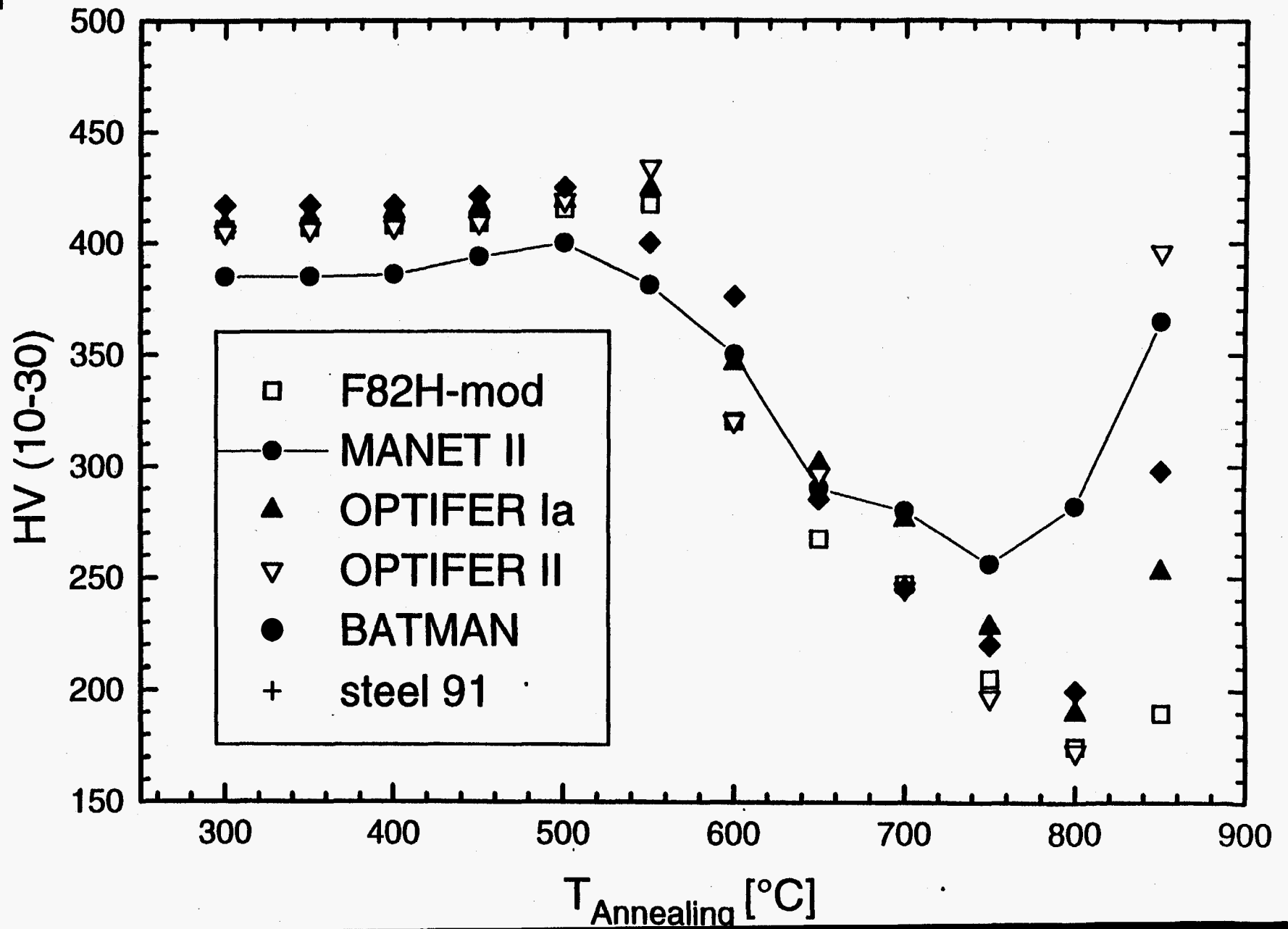
# Hardening Behaviour



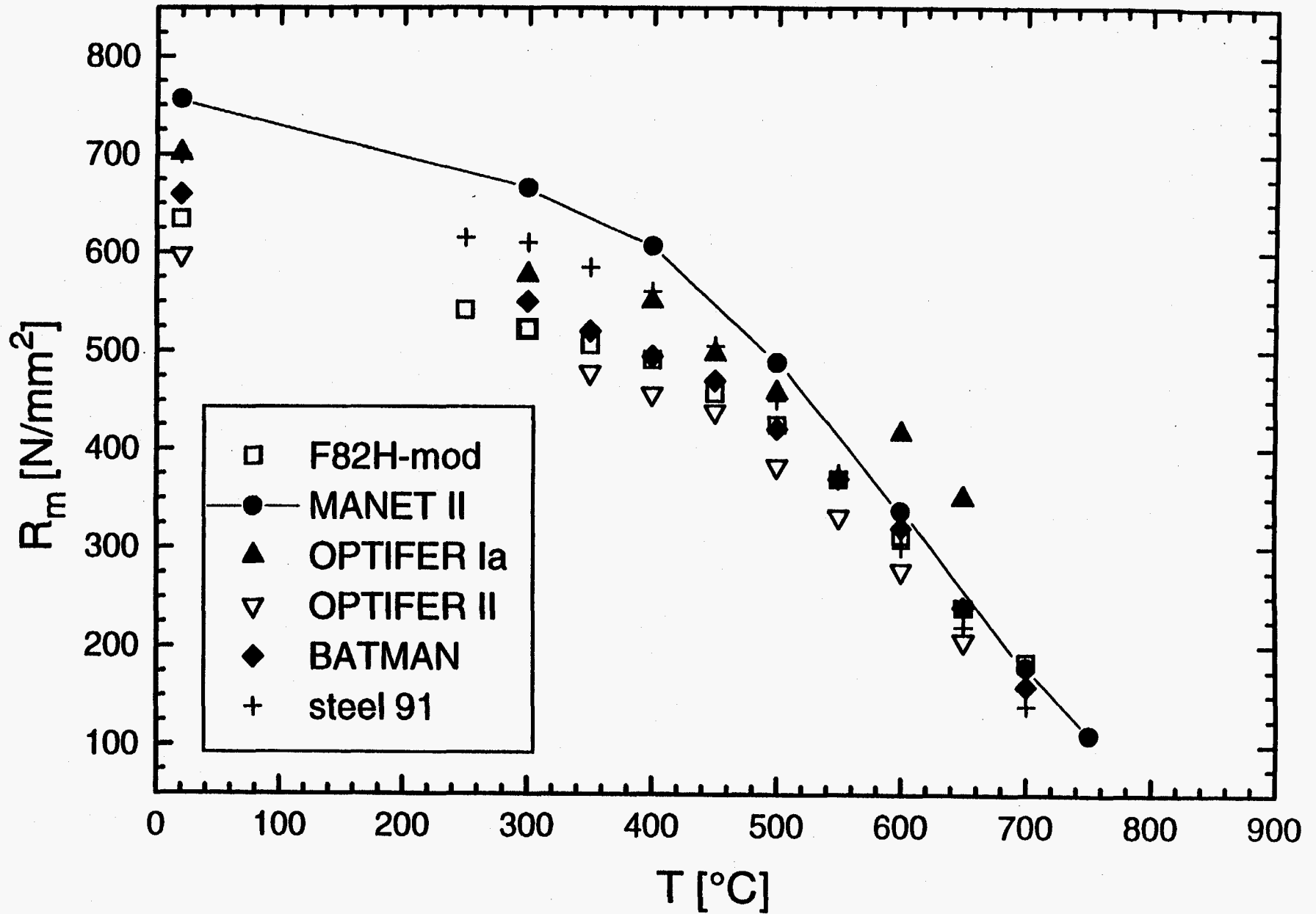
# Grain Size vs. Hardening Temperature

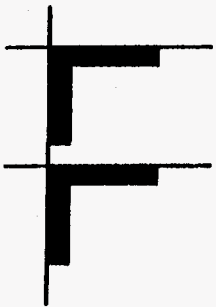


# Annealing Behaviour

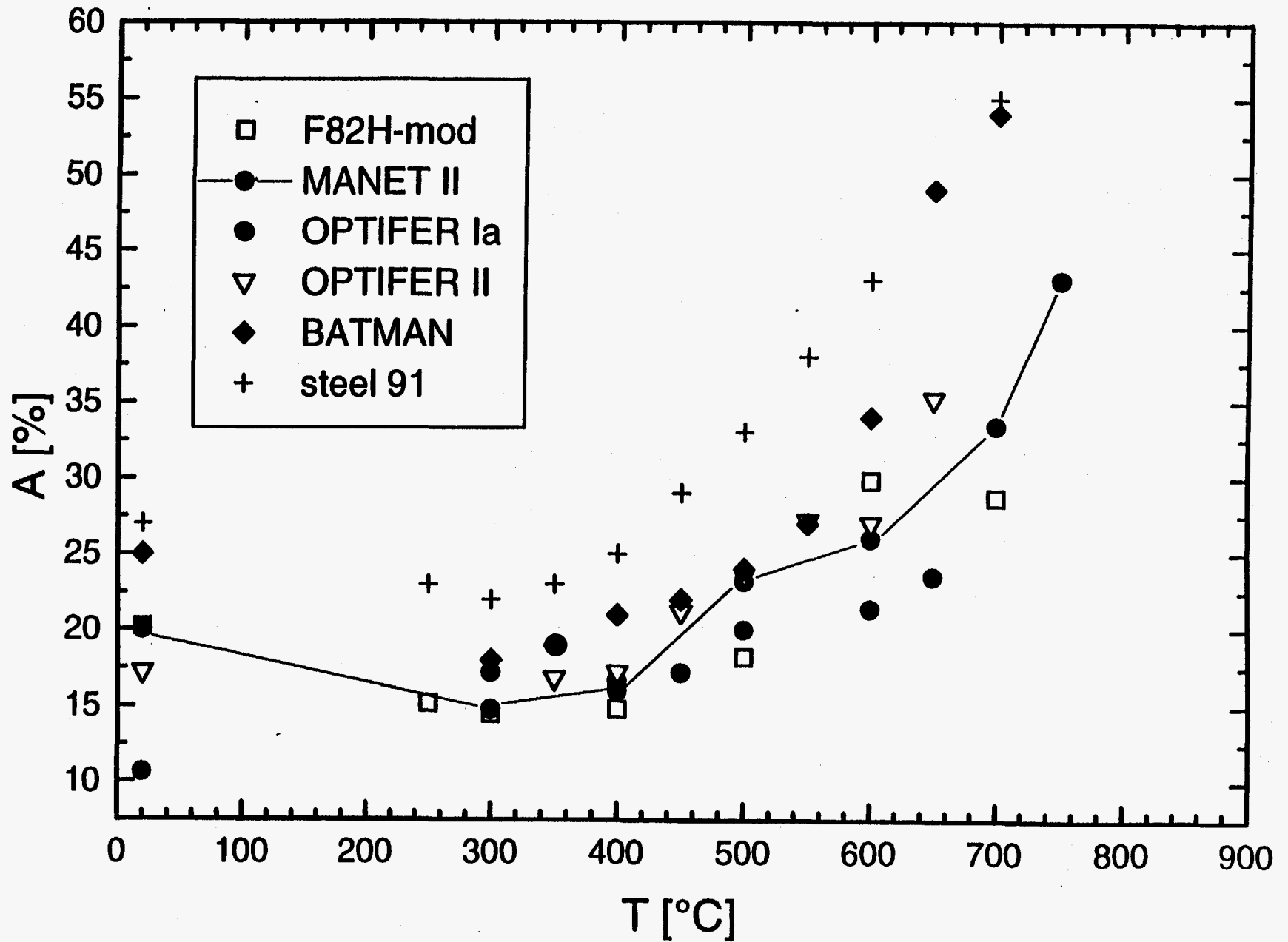


# Ultimate Tensile Strength

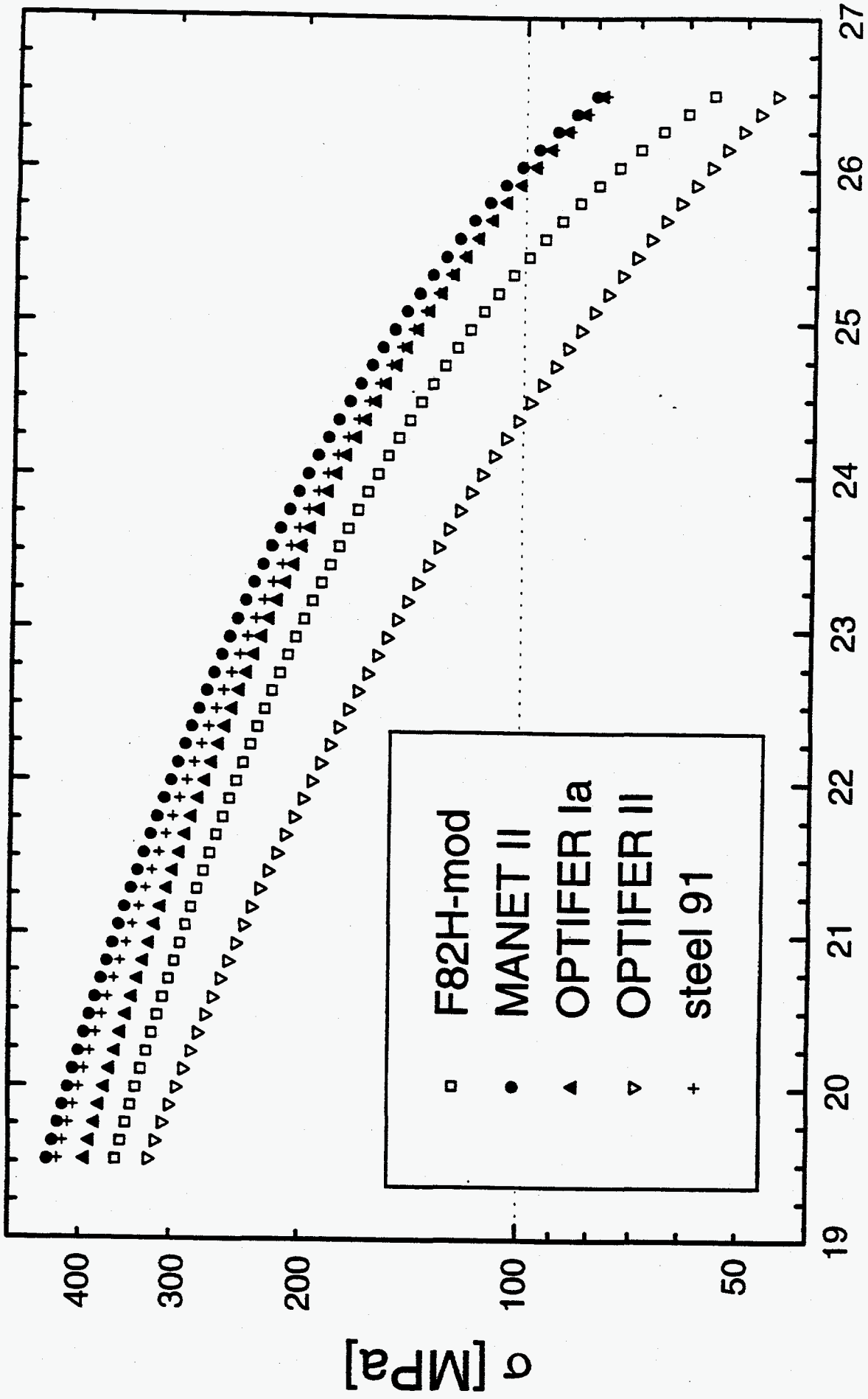




# Total Elongation



(Larsen Miller)

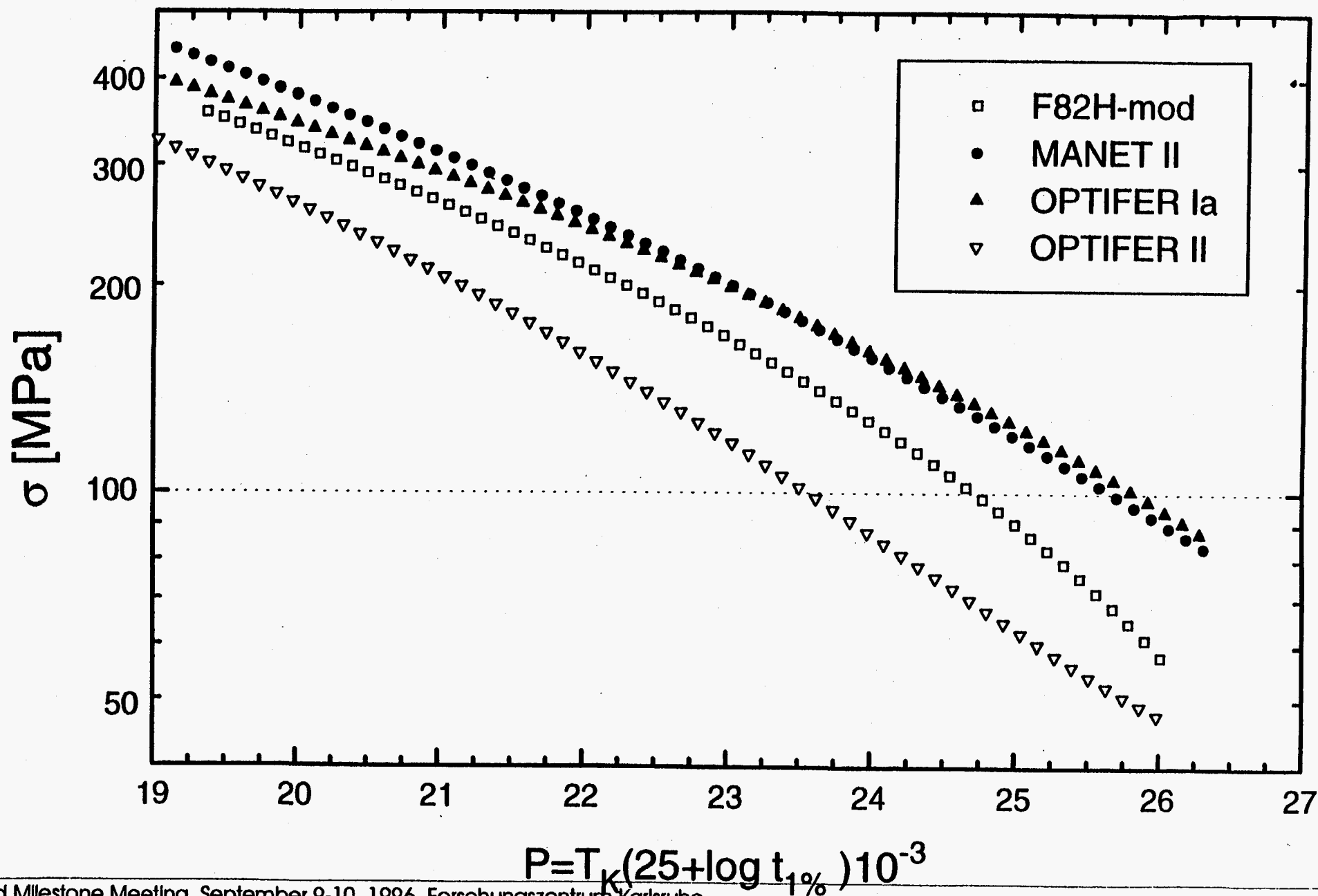


□ F82H-mod  
● MANET II  
▲ OPTIFER Ia  
▼ OPTIFER II  
+ steel 91

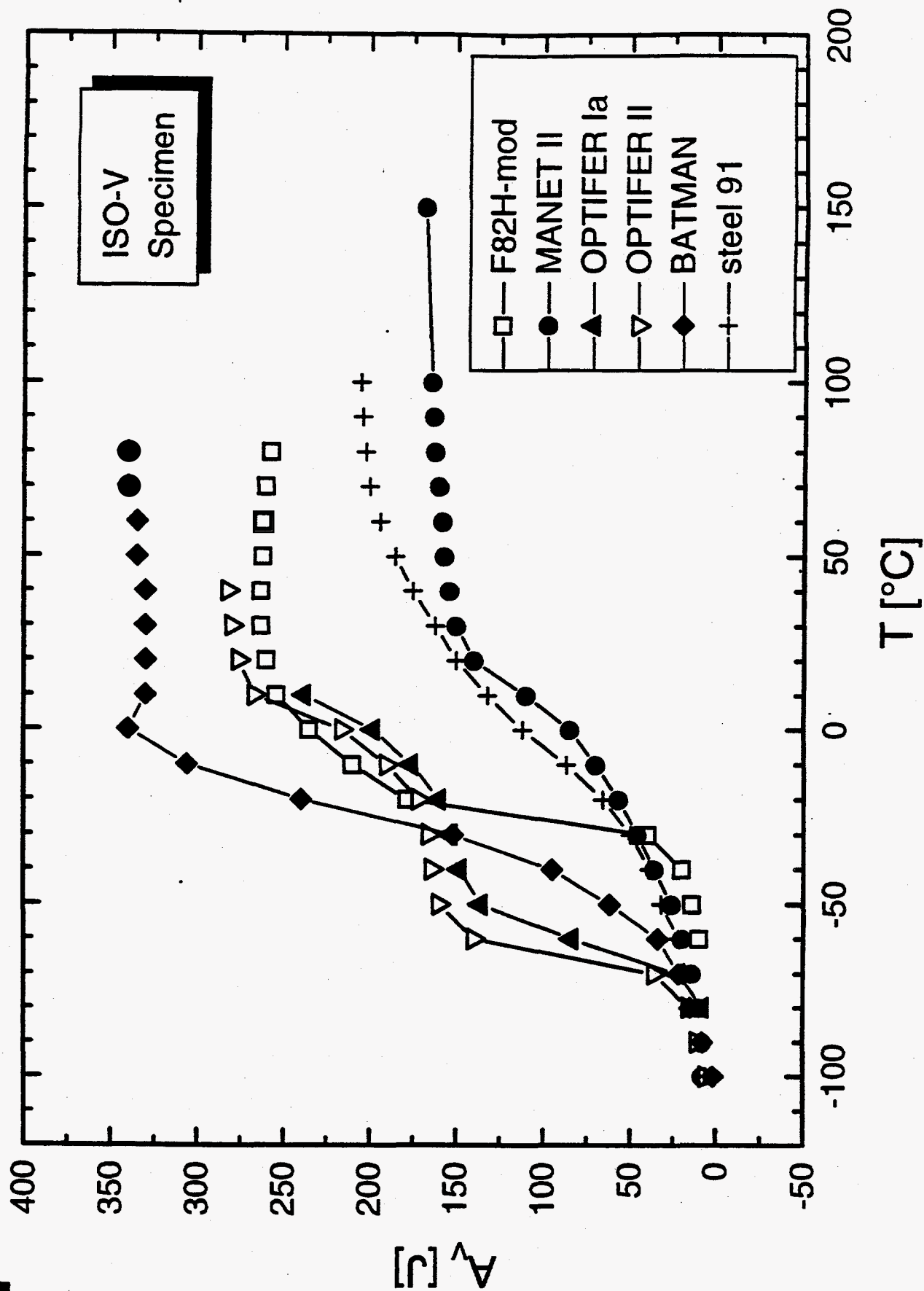
$$P = T_K(25 + \log t_R) \cdot 10^{-3}$$



# Master Plot for 1% Creep-Strain (Larsen Miller)



# Absorbed Energy vs. Temperature

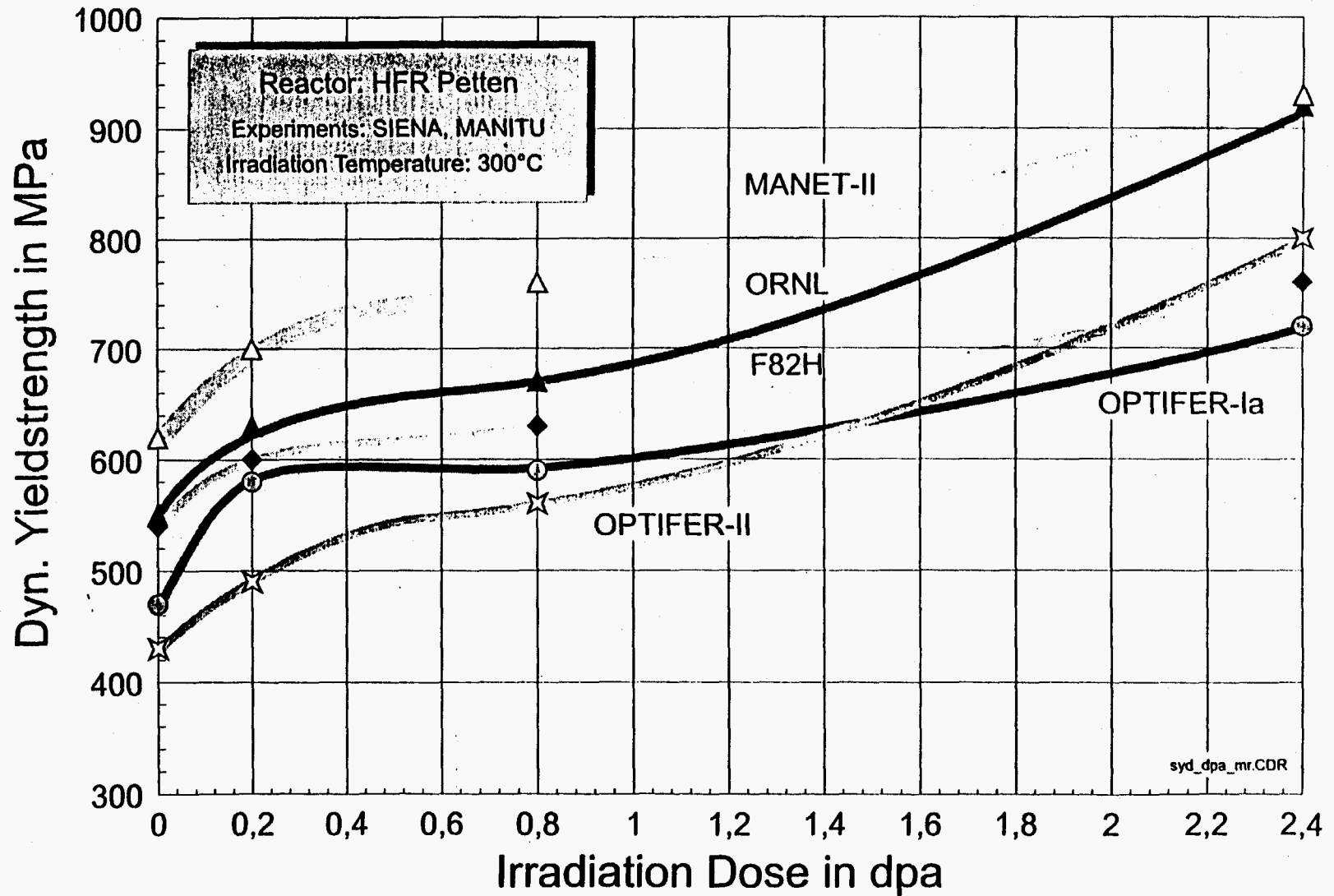


# Forschungszentrum Karlsruhe

Technik und Umwelt

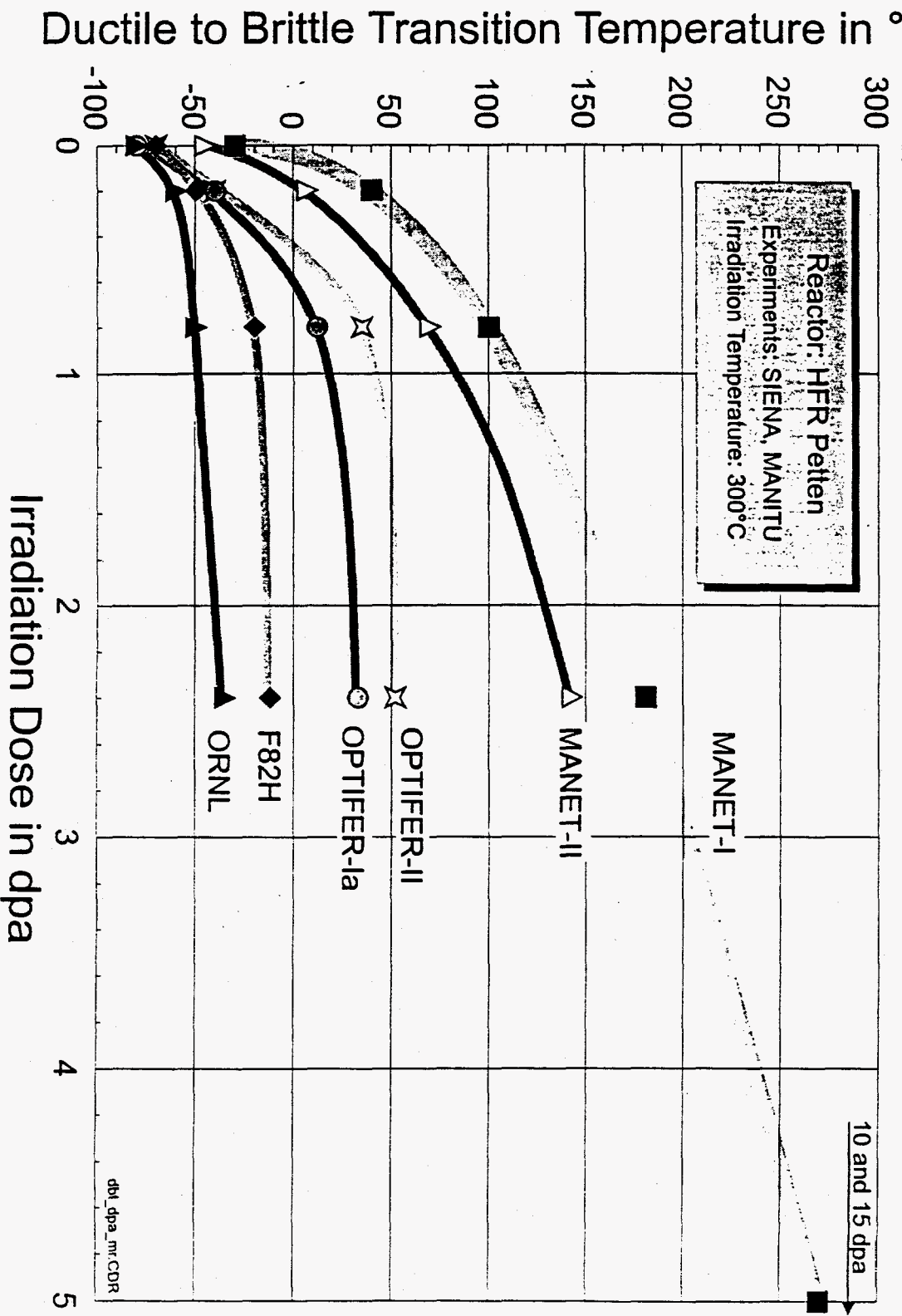
Institut für Materialforschung II

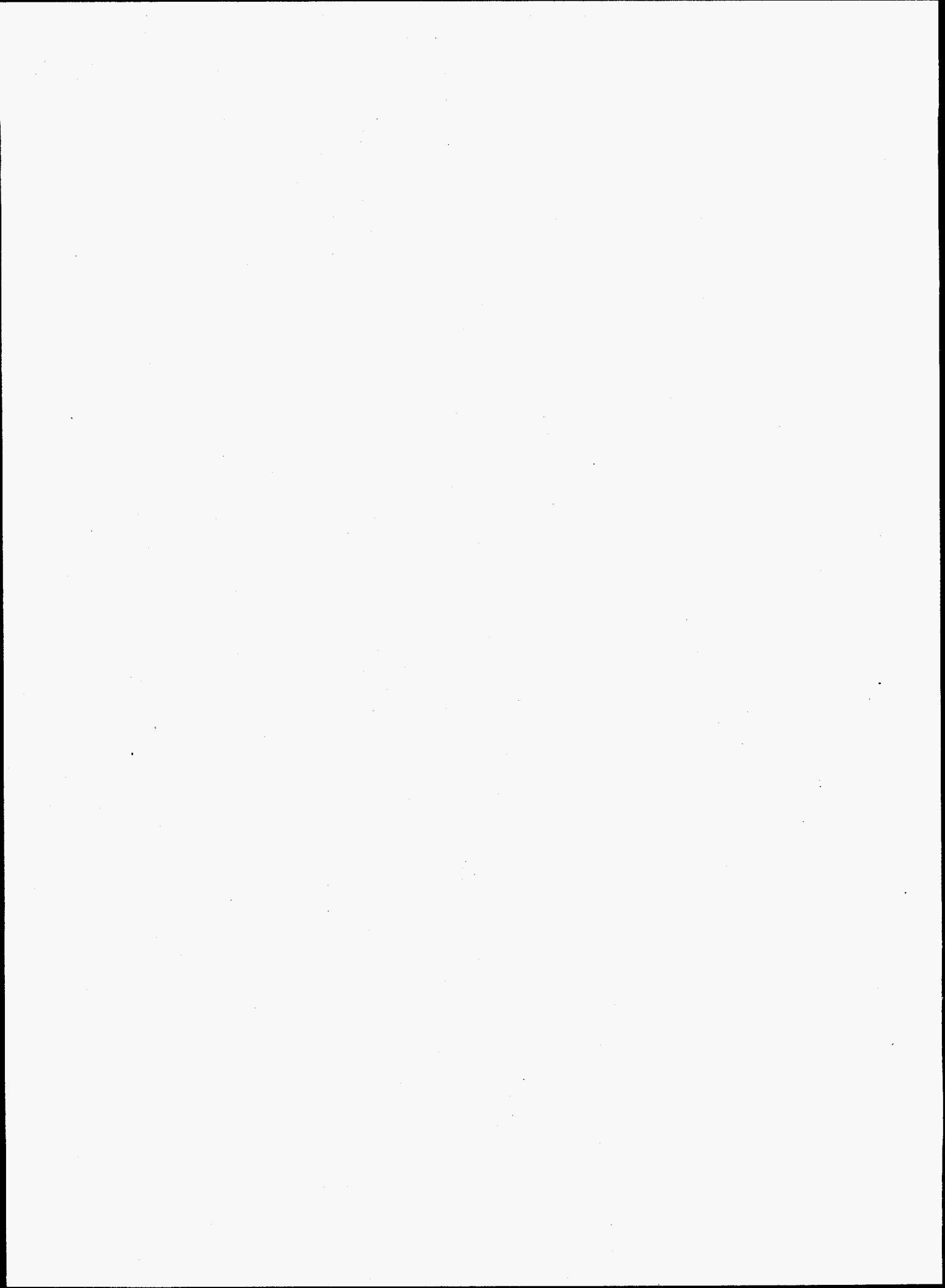
Leiter: Prof. Dr. D. Munz



# Forschungszentrum Karlsruhe Technik und Umwelt

Institut für Materialforschung II  
Leiter: Prof. Dr. D. Munz







*IEA Workshop and Working Group Meeting  
on Ferritic Steels  
JET Headquarters, Culham, UK  
24-25 October, 1996*

## **Summary of the Irradiation Data on JLF-1**

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# FFTF/MOTA IRRADIATION PROGRAM SUMMARY

(10/1/94)

Calendar Year	1987	1988	1989	1990	1991	1992	1993	1994	1995
Japanese Fiscal Year	S62	S63	H1	H2	H3	H4	H5	H6	H7
Original Plan (1987. 8. 14)	10 10/8	11 8/9	12 11/21	13 10/24	14 2/6	15 12/9			
Achievements	MOTA 1F 10 11/18 335EFPD 291MW	MOTA 2A 11 1/8	MOTA 2B 12 1/4	EBR-II COBRA-1A1 3/19 300EFPD	COBRA-1A2 12 5/27 203EFPD	COBRA-1A1 11/27 88.5EFPD	COBRA-1A2 4/16 337.3EFPD	HFIR (JP-23) 9/26 12/16 110.2EFPD	

## FFTF/MOTA Irradiation Condition

### FFTF Cycle11 (MOTA 2-A)(299.7EFPD)

Temp (C)	Dose (dpa)	MOTA Canister
375	12	BC-E
390	31	1A-3
410	36	2D-3
460	31	1F-2
520	34	4E-4
600	36	3F-3

### FFTF Cycle12 (MOTA 2-B)(203.3EFPD)

Temp (C)	Dose (dpa)	MOTA Canister
375	8	BC-E
390	21	1A
410	24	2E
460	21	1F
520	23	4E
600	24	3F



= EBR-II irradiation condition =

COBRA 1A1 (Cold B7A Radiation Assembly)

Run 162 & 163 (96/11/26~93/4/1) (88.6EFPD, ~8dpa)

673K	773K	873K
$1.71 \times 10^{22} \text{n/cm}^2$	$1.66 \times 10^{22} \text{n/cm}^2$	$1.66 \times 10^{22} \text{n/cm}^2$
8.2dpa	8.0dpa	8.0dpa

JLF-1, JLF-3, JLF-4, F82H, JFMS, HT9

Tensile(S), 1.5CVN, 1/3CVN, TEM

= PIEs =

Tensile tests using MATRON (Irrad. temp., Room temp.)

Charpy test (1.5mm, 1/3 size)

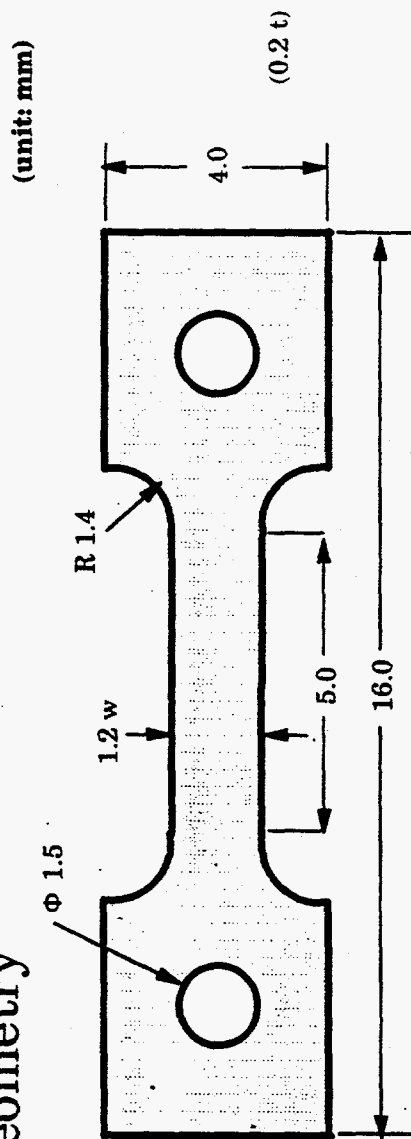
Disk bend tests

TEM

Precipitation analyses

# Tensile(S)/MATRON

## Tensile(S) Geometry



## MATRON Tensile Test Condition

Test temperature: Irradiation temperature

Room temperature

Vacuum:  $(1 \sim 3) \times 10^{-6}$  Torr.

Strain rate:  $1 \times 10^{-3} \text{ sec}^{-1}$

## Mini-size Charpy Tests (1.5mm size)

---

JLF-1 (9Cr-2W)

F82H (8Cr-2W)

JLF-3 (7Cr-2W)

---

573K (300C) / 0.01dpa  
(JMTR)

693K (420C) / 35dpa  
(FFTF/MOTA-2A)

693K (420C) / 35dpa  
(FFTF/MOTA-2A)

648K (375C) / 20dpa  
(FFTF/MOTA-2A & 2B)

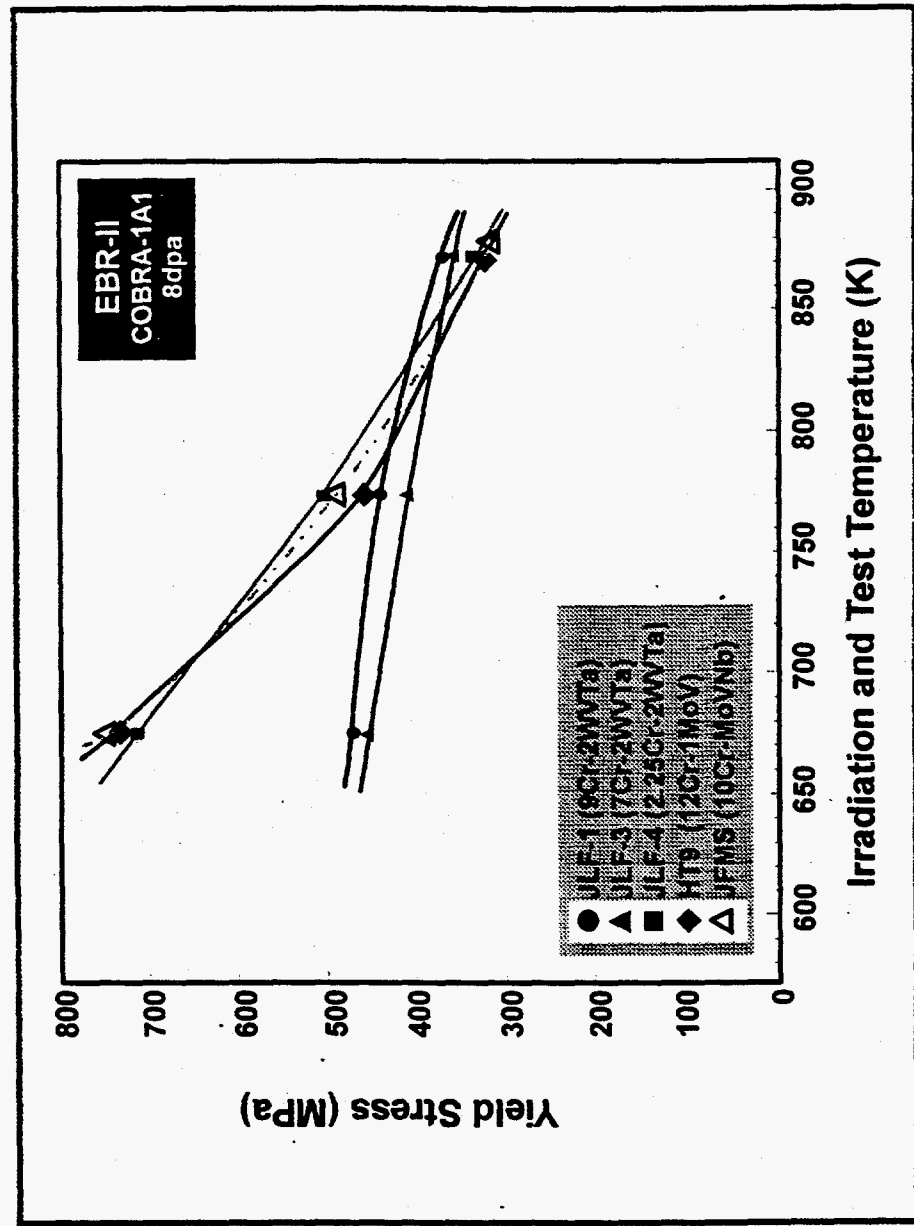
678K (405C) / 35dpa  
(FFTF/MOTA-2A)

683K (410C) / 35dpa  
(FFTF/MOTA-2A)

683K (410C) / 60dpa  
(FFTF/MOTA-2A & 2B)

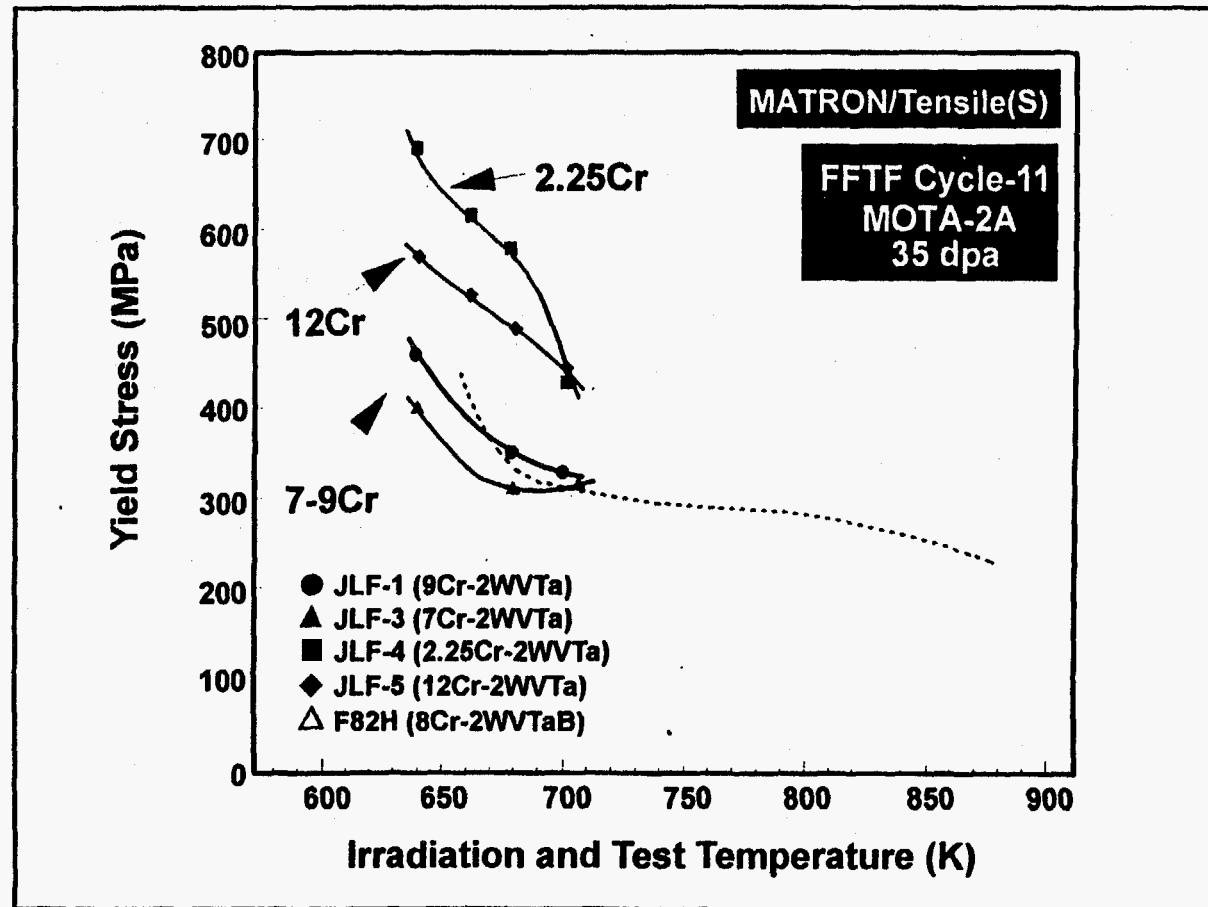
---

# EBR-II / MATRON / Tensile(S)



## Tensile Test Results / MATRON / Tensile(S)

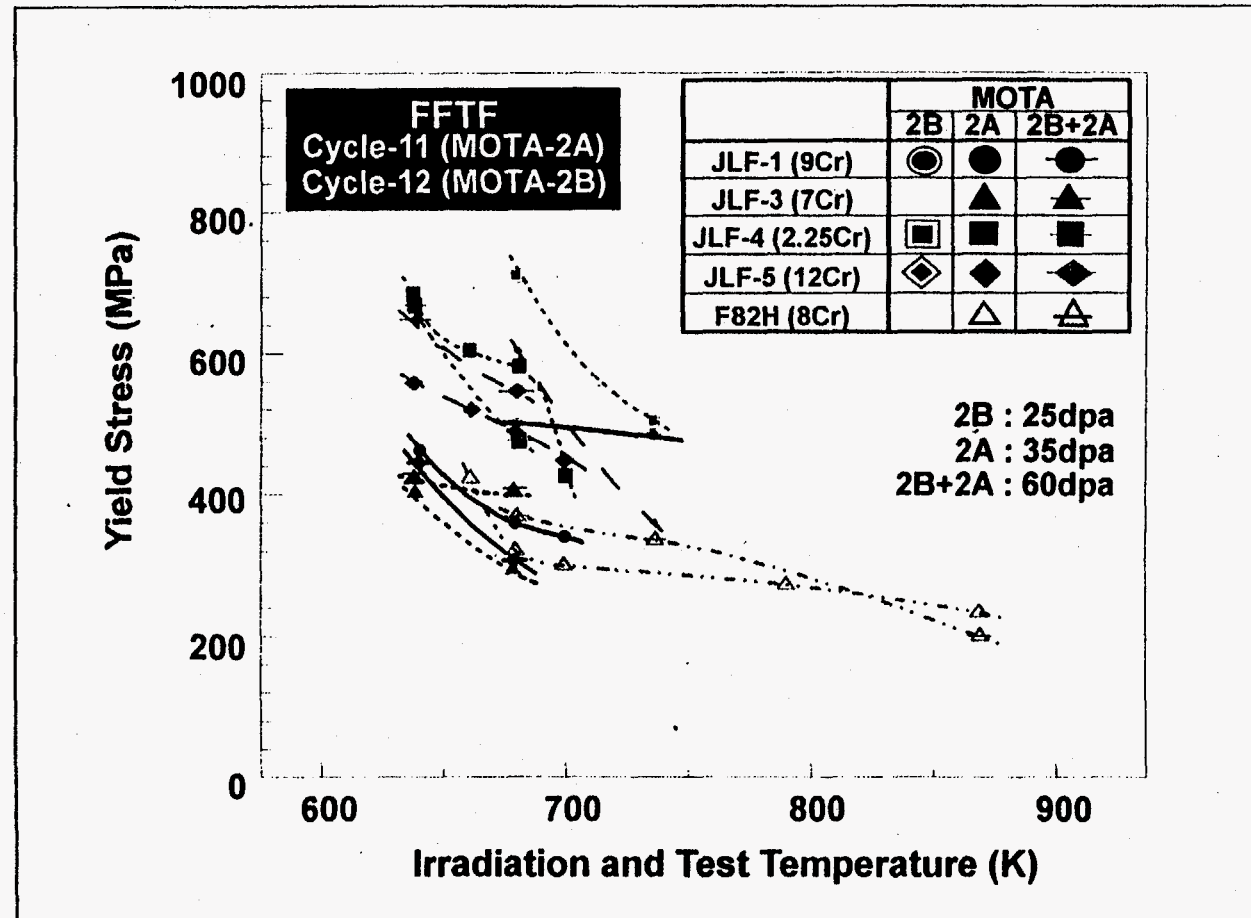
Temperature dependence of yield strength following irradiation to 35dpa in the FFTF/MOTA



■ Essentially same temperature dependence and Cr level dependence of yield stress

## Tensile Test Results / MATRON / Tensile (S)

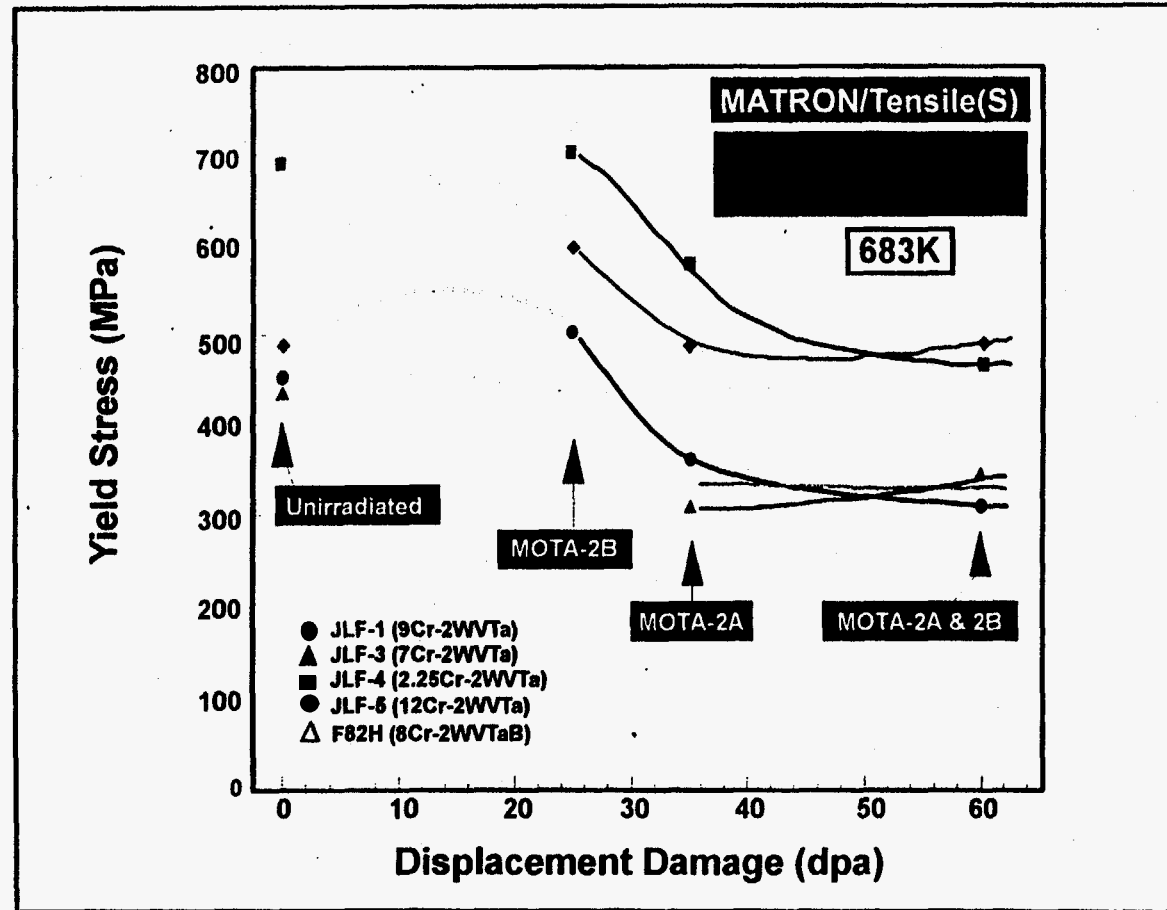
Irradiation temperature and dose dependence of yield strength of JLF series steels and F82H irradiated in the FFTF/MOTA



- Possible to classify into two groups; 7-9Cr steels & 2.25Cr, 12Cr steels
- Yield strength range at 683K; 300-400MPa (7-9Cr steels), 450-700MPa (2.25Cr, 12Cr steels)

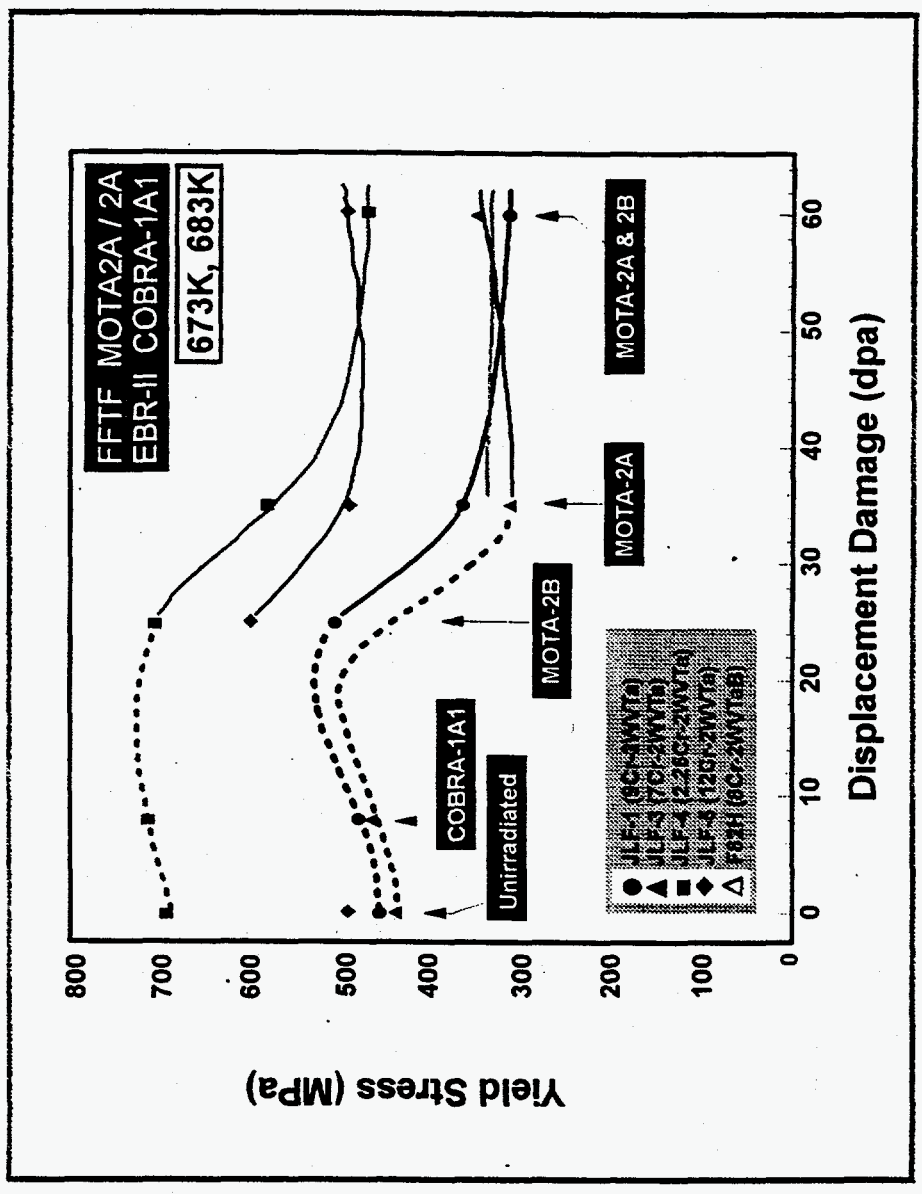
## Tensile Test Results / MATRON / Tensile (S)

Dose dependence of yield strength following irradiation at 683K  
in the FFTF/MOTA



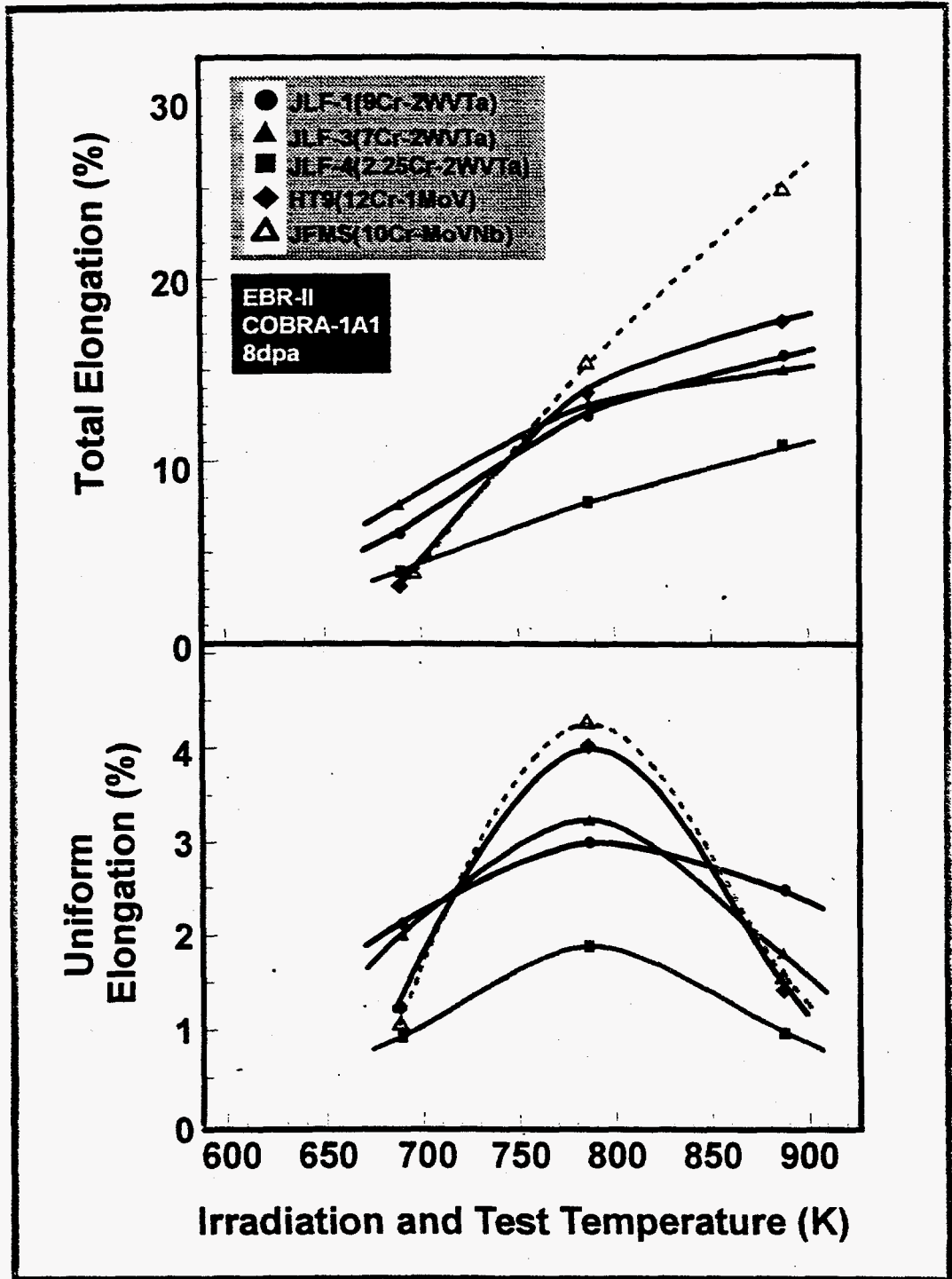
- Yield strength once increases with irradiation dose and then decreases to become saturation level over 35-40dpa. Peak hardening at around 10-15dpa (?)
- Lower saturation strength level in 7-9Cr steels than 2.25Cr, 12Cr steels

# Tensile Test Results / MATRON / Tensile (S)



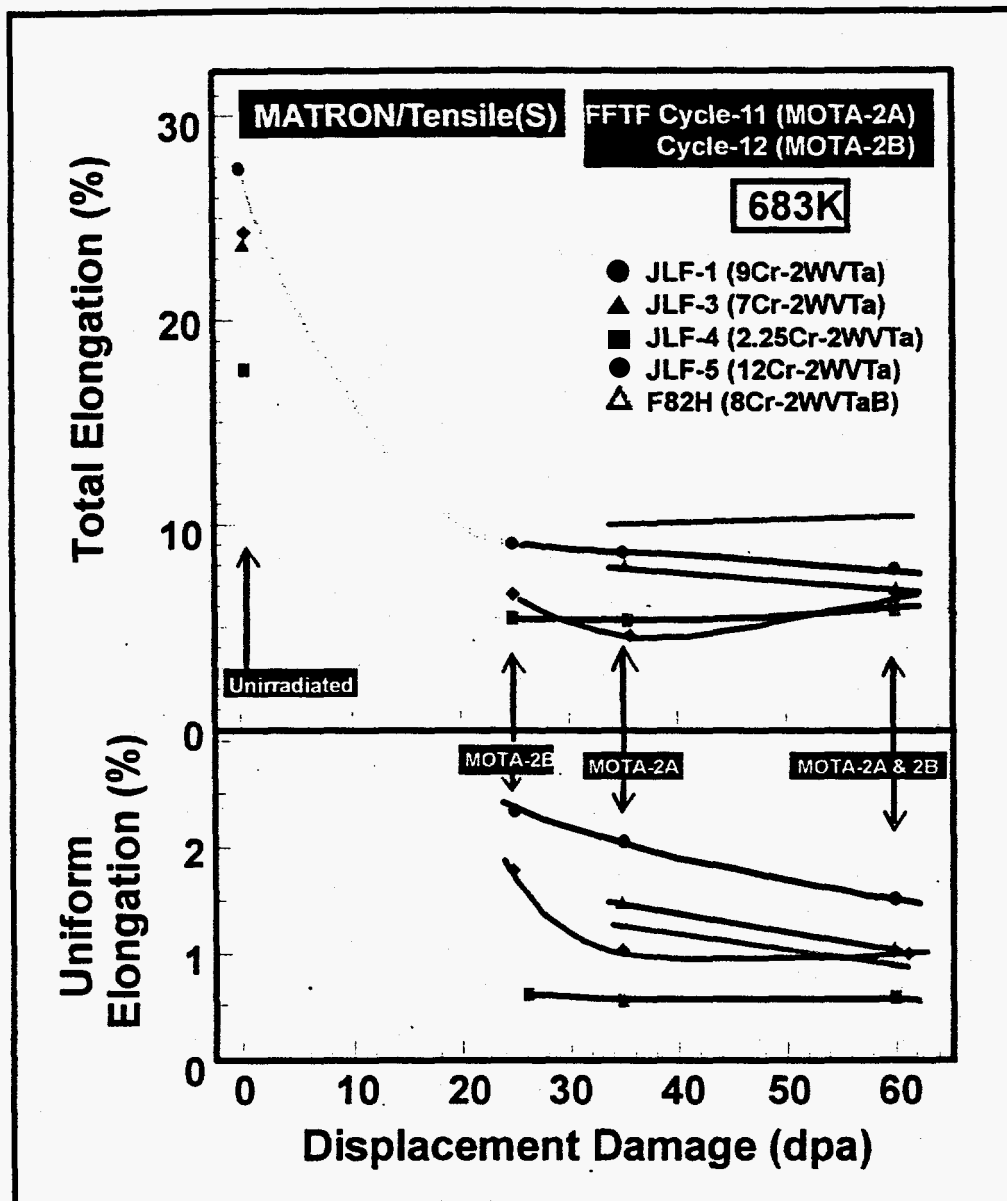


# EBR-II / MATRON / Tensile (S)



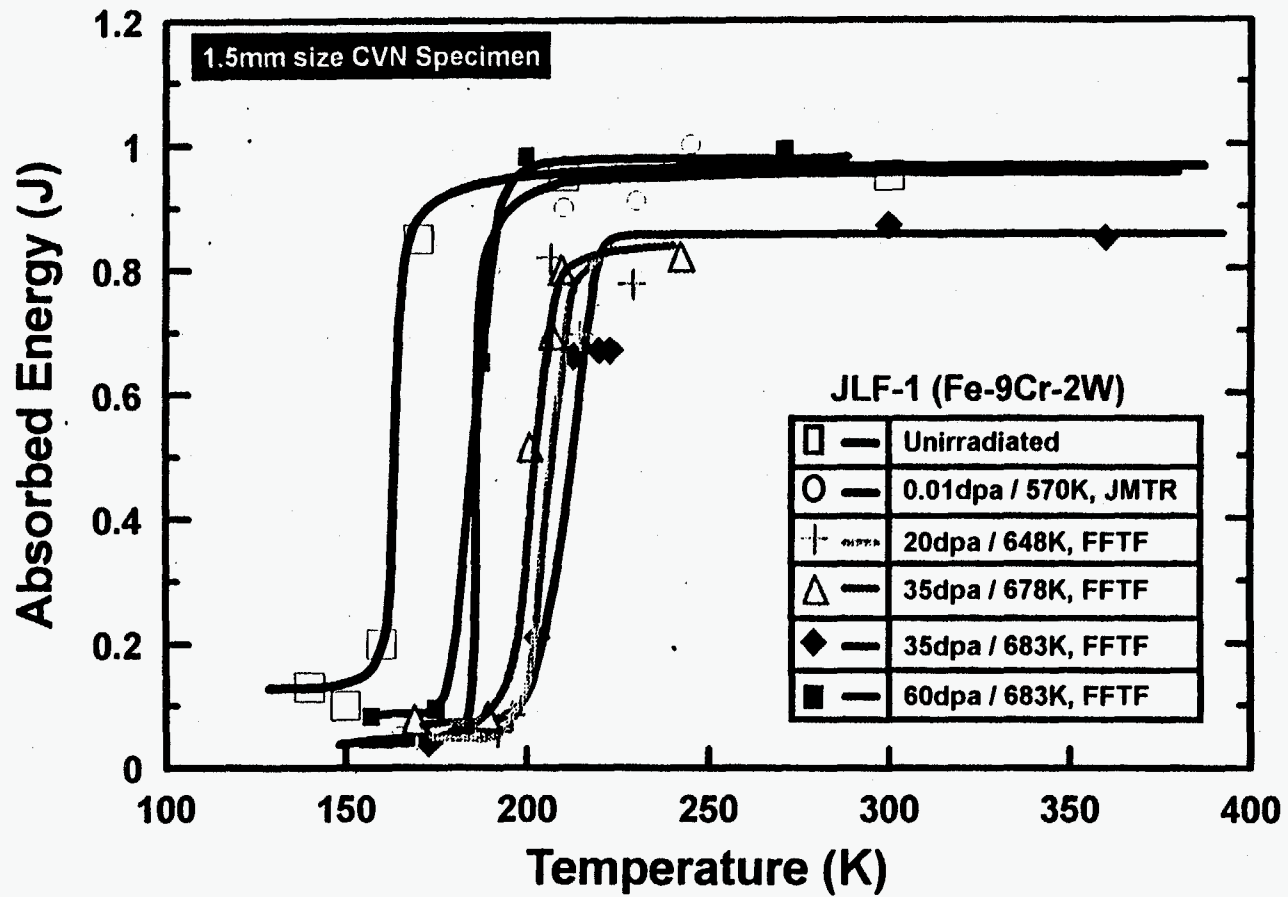
## Tensile Test Results / MATRON / Tensile (S)

Dose dependence of uniform and total elongation in JLF series steels and F82H following irradiation at 683K in the FFTF/MOTA



- Relatively larger total and uniform elongation in 7-9Cr steels than in 2.25Cr, 12Cr steels
- Elongation seems to reach saturation over about 35dpa.

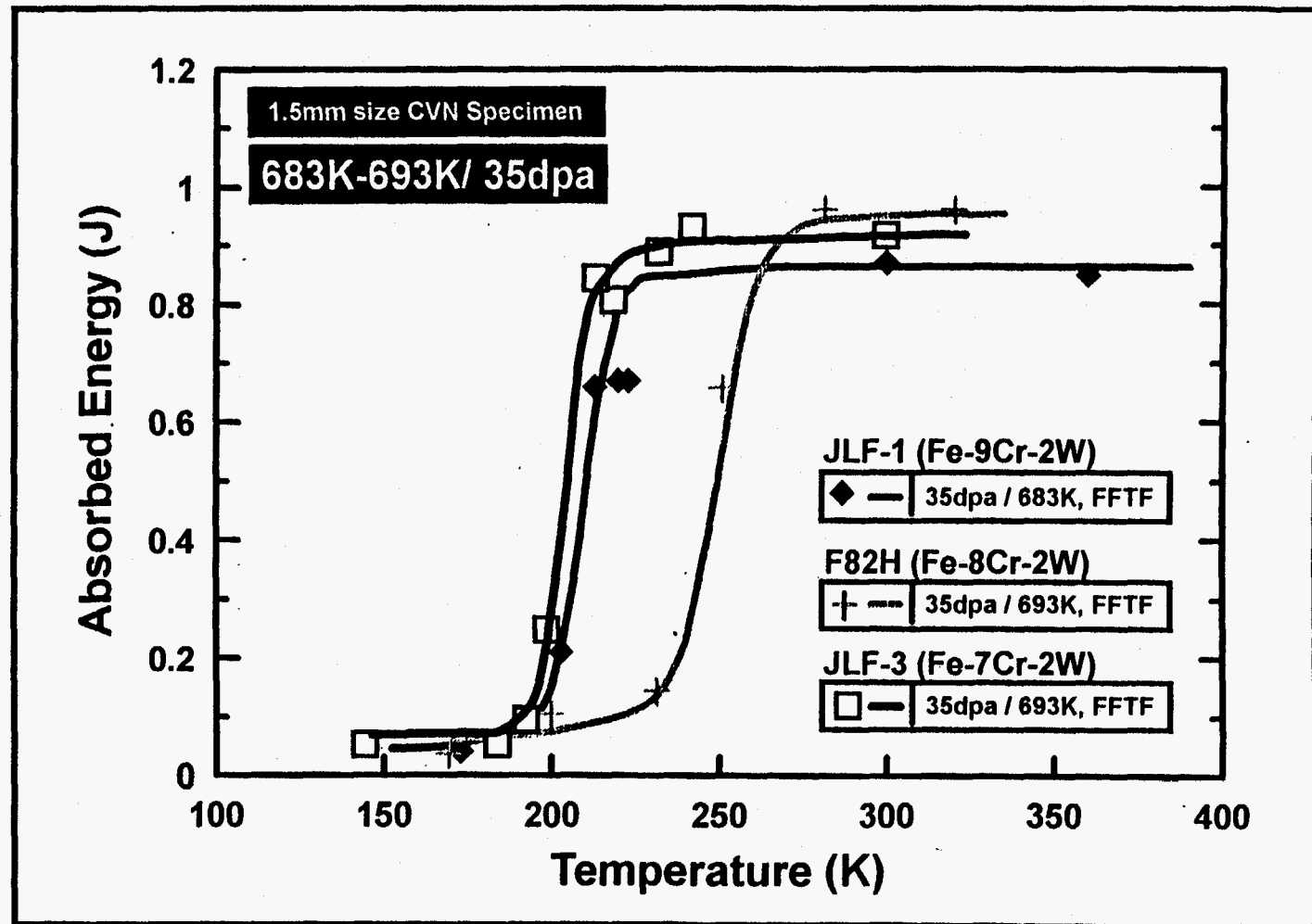
# Charpy Impact Test Results = JLF-1 = (573K - 683K : 0.01dpa - 60dpa)



# Charpy Impact Test Results / 1.5mm CVN

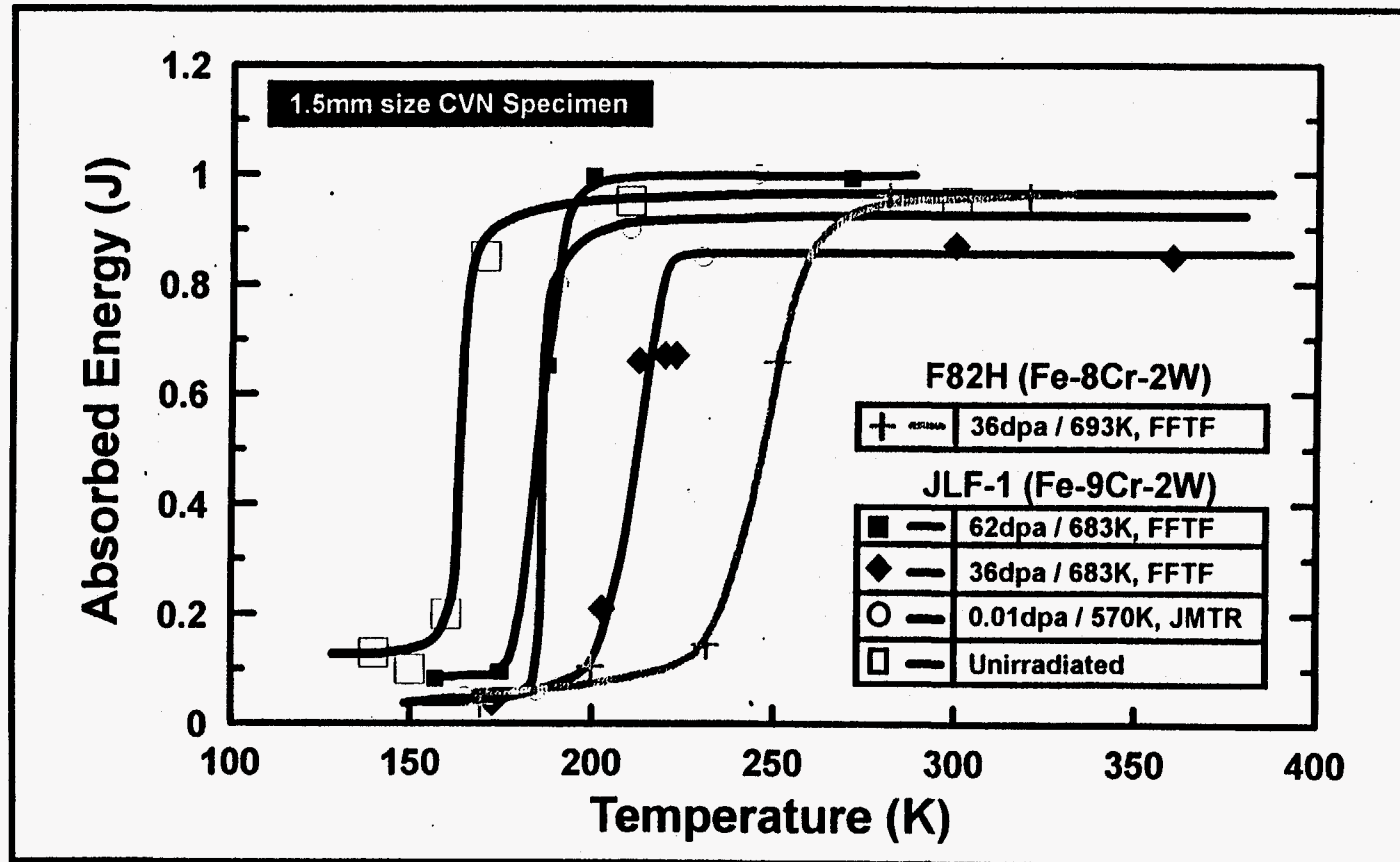
= JLF-1, F82H, JLF-3 =

(683K-693K: 35dpa)



## Charpy Impact Test Results / 1.5mm CVN

Charpy impact curves for JLF-1 (Fe-9Cr-2W) and F82H (Fe-8Cr-2W) following irradiation to 0.01-60dpa at 570-693K in JMTR or FFTF



- Excellent Charpy impact response in JLF-1 and F82H, DBTT still remains below R.T.
- Superior DBTT property of JLF-1 than F82H
- DBTT decreases at high displacement damage level (in JLF-1)

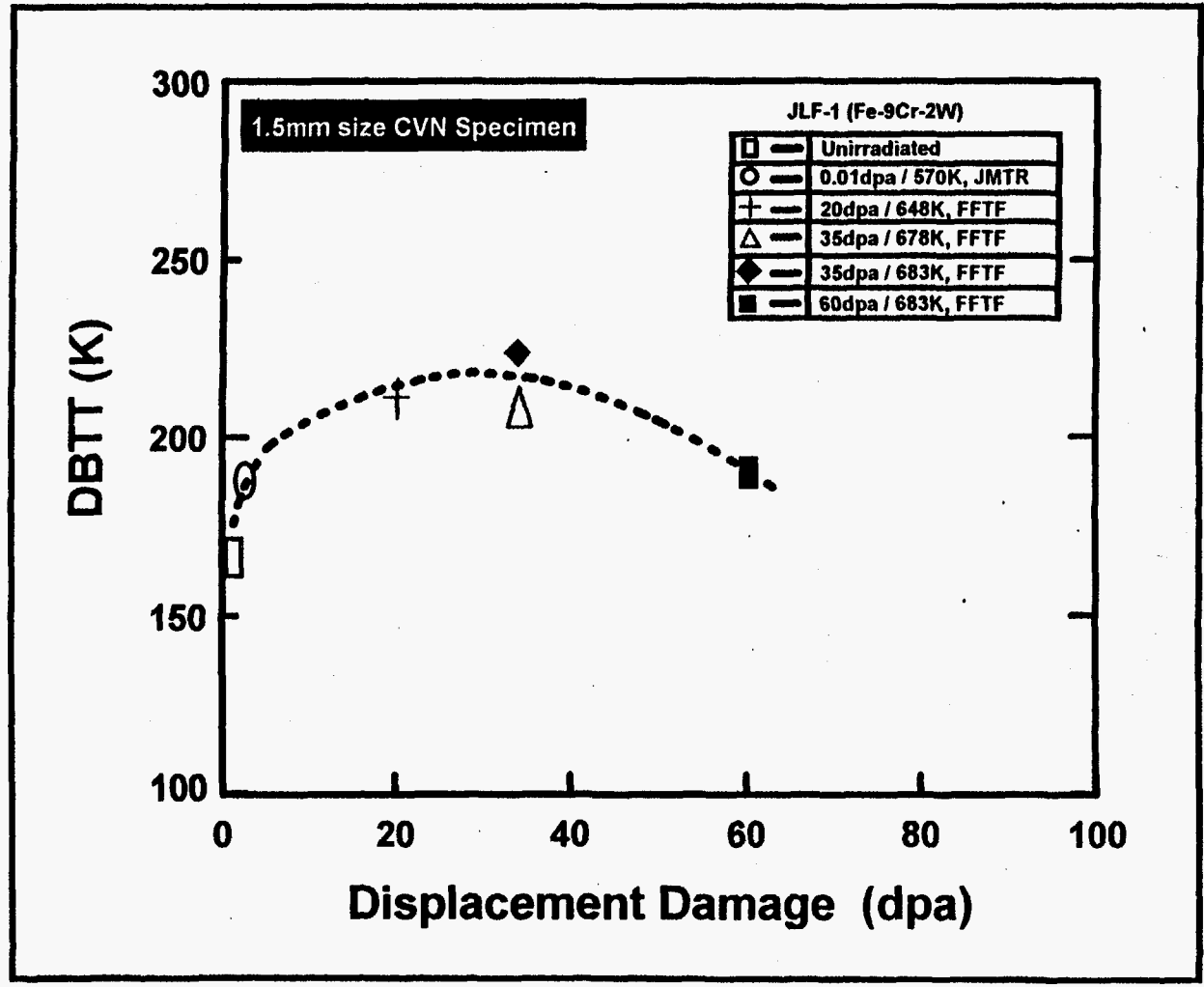
# Material

(mass%)

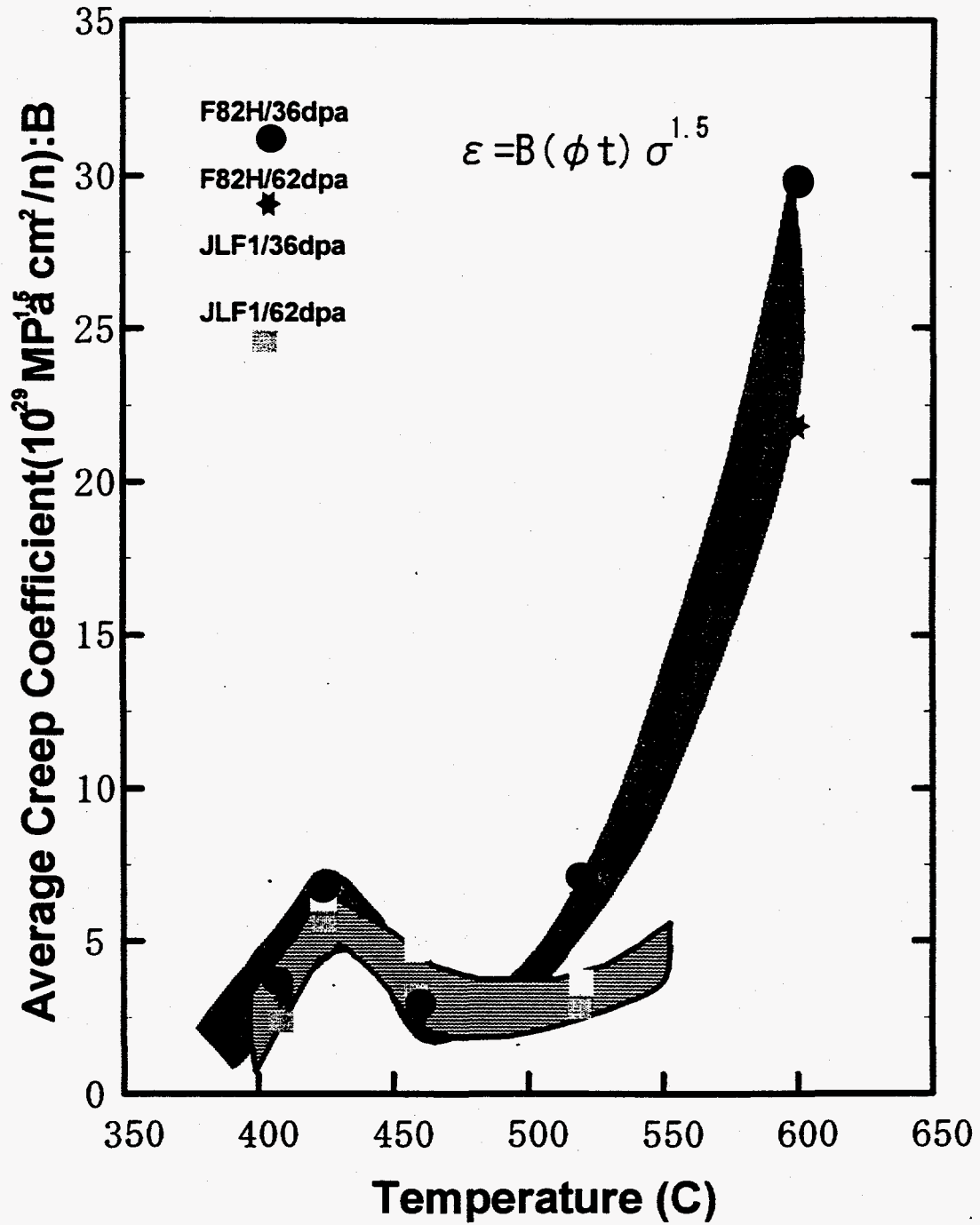
	C	Si	Mn	Ni	Cr	Mo	V	Nb	Ti	W	Ta	B
JLF1	0.097		0.46		9.04		0.19	-	0.001	1.97	0.070	-
JLF2	0.095		0.45		9.16		0.20	-	0.010	1.93	0.072	-
JLF3	0.093		0.45		7.03		0.20	-	0.001	1.97	0.070	-
JLF4	0.101	0.07	0.50		2.23		0.20	-	0.003	1.97	0.068	-
JLF5	0.094		0.48		11.99		0.19	-	0.002	1.98	0.066	-
JLF6	0.096		0.46		12.00		0.19	-	0.010	1.94	0.068	-
F82H	0.093	0.09	0.49	0.01	7.65	0.00	0.18	0.00	-	2.0	0.040	0.0034
HT-9	0.21	0.21	0.50	0.57	12.10	1.03	0.33	-	-	0.52	-	-
JFMS	0.050	0.67	0.58	0.94	9.85	2.31	0.12	0.06	-	-	-	-

JLF-1,2,3,5,6: 1050C x 0.5hr (normalizing) + 775C x 1hr (tempering)  
 JLF-4: 1050C x 0.5hr (normalizing) + 700C x 1hr (tempering)

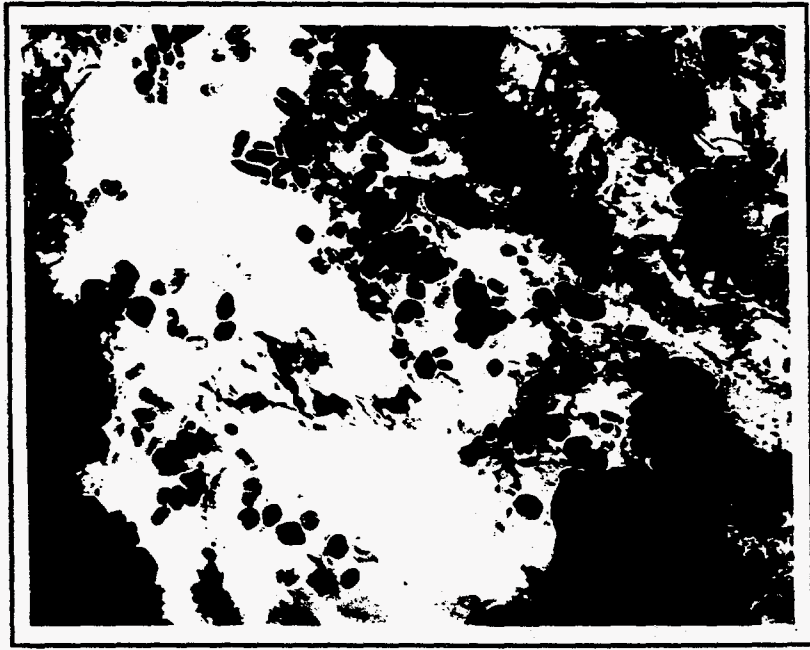
**Charpy Impact Test Results**  
**= JLF-1 =**  
**(573K - 683K : 0.01dpa - 60dpa)**



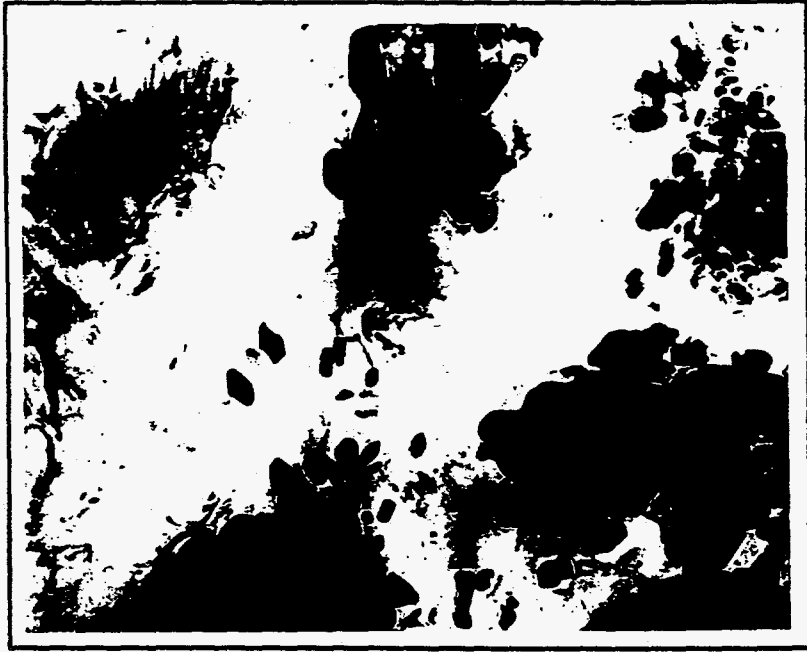
# TEMPERATURE DEPENDENCE OF AVERAGE CREEP COEFFICIENT







673K



200nm

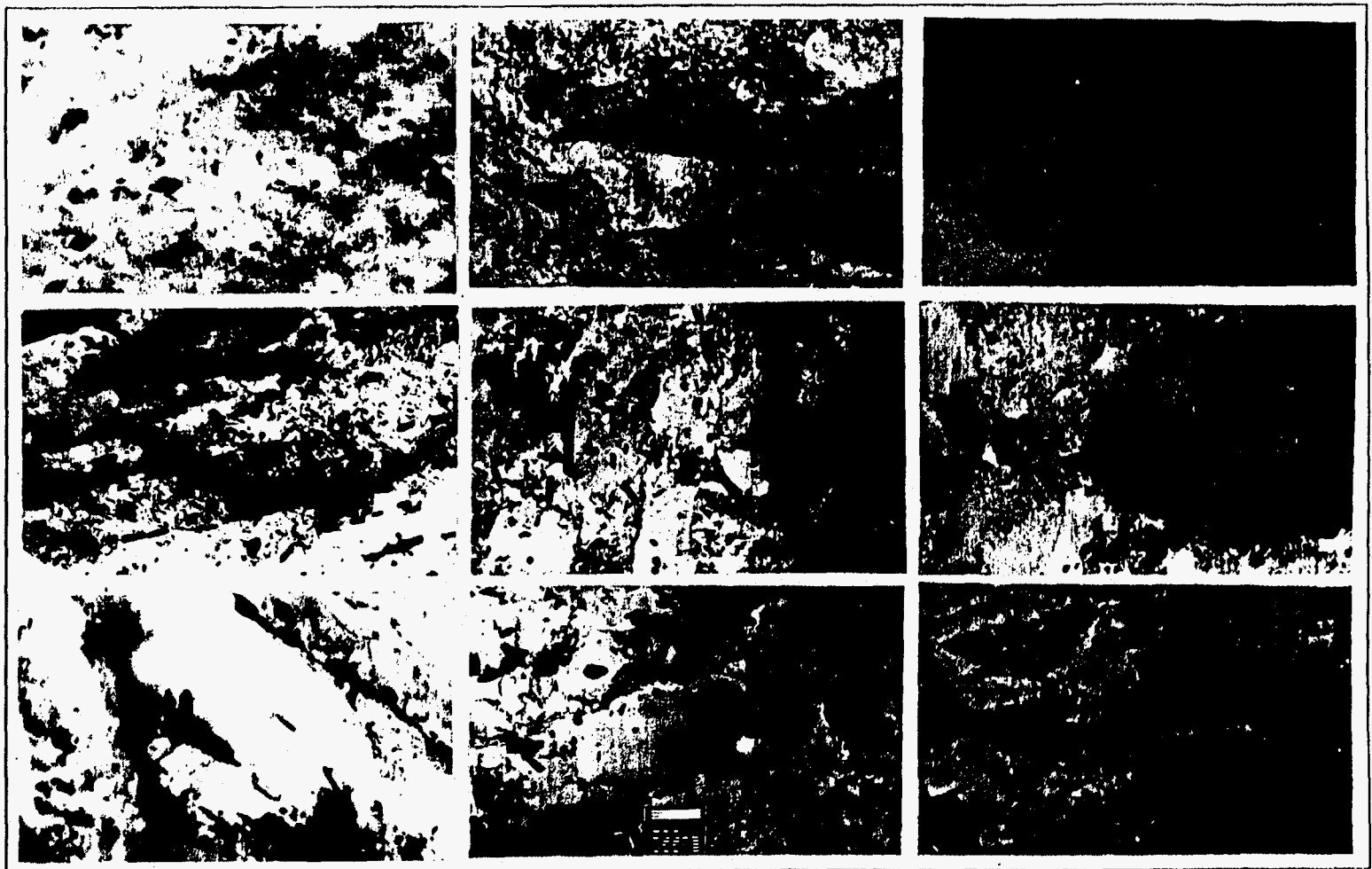
773K

Precipitates in F82H irradiated to 8dpa in EBR-II (COBRA-1A1)

663K

683K

733K



JLF-1 (9Cr)

JLF-3 (7Cr)

JLF-5 (12Cr)

Microstructures of JLF-1, JLF-3 and JLF-5 following irradiation to 35dpa at 663K, 683K and 733K at FFTF/MOTA

JLF1



673K

JLF3



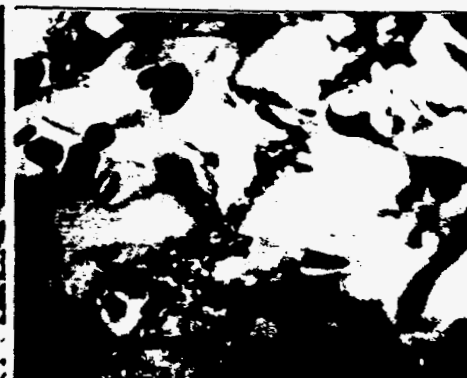
JLF4



F82H



773K



100nm

Microstructure of JLF1, JLF3, JLF4 and F82H irradiated to 8dpa  
in EBR-II(COBRA-1A1)

## **Irradiation Effects on Mechanical Properties of Japanese RAFs (FFTF/MOTA Irradiation)**

### **Miniaturized Charpy Results:**

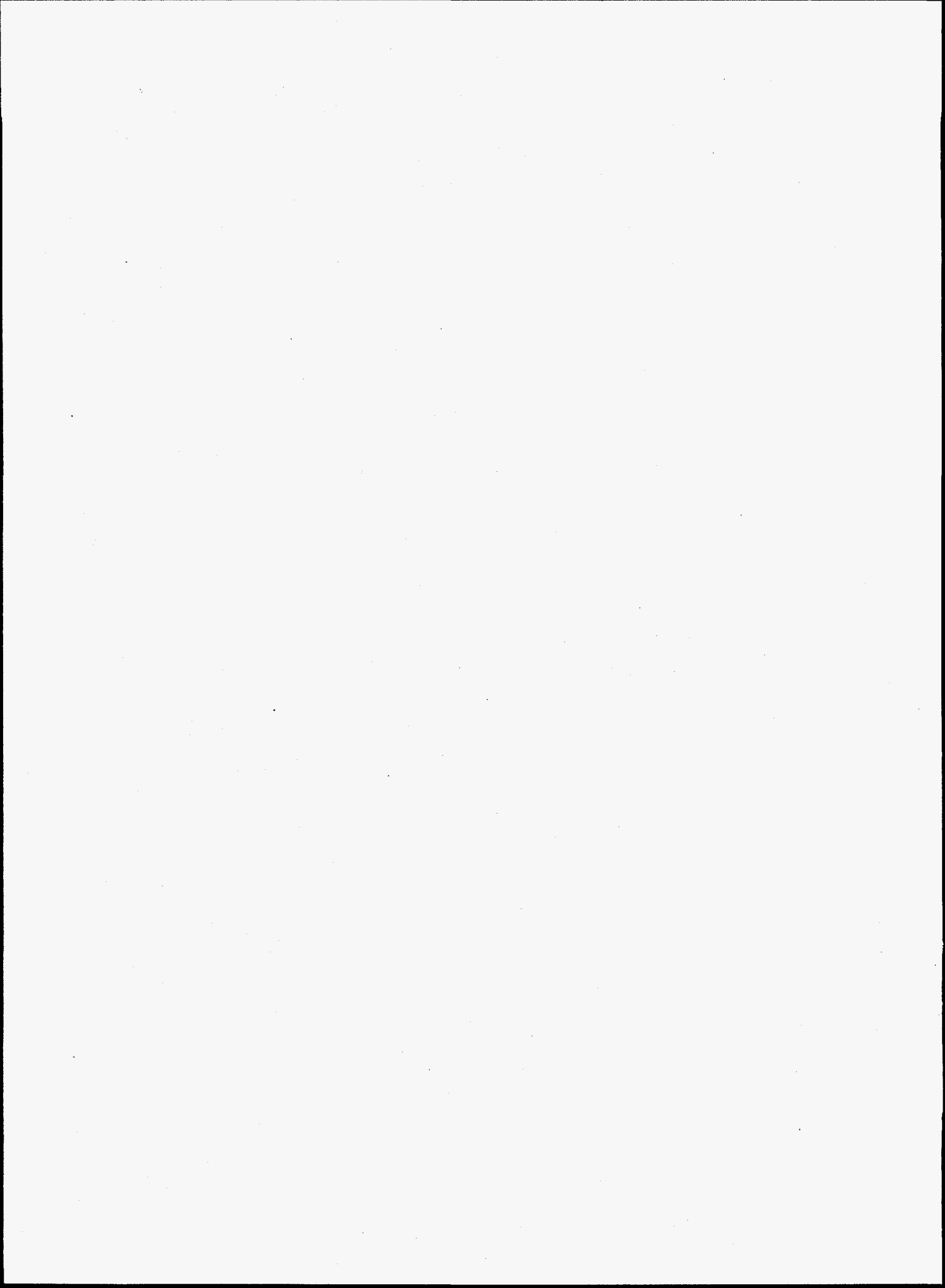
<b>JLF-1</b>	<b>Increase of DBTT :</b>	<b>0 - 35dpa (50K)</b>
	<b>Decrease of DBTT:</b>	<b>35 - 60dpa</b>
	<b>Decrease of USE at 35dpa:</b>	<b>10%</b>
<b>F82H</b>	<b>Larger DBTT shift (increase) than JLF steels (JLF-1) 40K (35dpa)</b>	

### **Miniaturized Tensile Results:**

- Peak hardening at around 20dpa**
  - 300-400MPa in (7-9)Cr steels**
  - 450-700MPa in 2.25Cr, 12Cr steels**
- Saturation of elongation at 25-60dpa**

### **Pressurized Tube Creep Results:**

- Creep coefficient peak at around 430K**
- Less creep resistivity in F82H at high temperature**



THE PROGRESS OF  
ROUND-ROBIN TEST  
IN JAPAN

K. Shiba  
JAERI

IEA Working Group Meeting  
on Ferritic Steels  
24-25, October, 1996

## JAPANESE IEA ROUND ROBIN TEST PROGRAM ON F82H

### 1. Metallurgical tests (Base Metal) (1995 - 1996)

Test	Schedule	Remarks
Macro/Micro structure	1995	Optical, SEM, TEM microscopy
Hardness	1995	
CCT curve	1995 - 1996	

### 2. Mechanical properties (Base Metal) (1995 - 1997)

Property	Test condition	Schedule	Remarks
Tensile	RT-600°C	1995 - 1996	
Charpy Impact	Full curve	1995 - 1996	
Fracture toughness( $K_{IC}$ , $J_{IC}$ )	RT, 100, -30°C	1995 - 1996	
Creep			
Creep Rapture	500 - 650°C	1995 - 1997	max: 50000h
Creep Rate	500 - 650°C	1995 - 1997	max: 3000h
Creep Curve	500 - 650°C	1995 - 1997	max: 3000h
Creep Fatigue	500 - 650°C	1995 - 1997	holding time: 0,1,3,10,30min
Fatigue	RT - 600°C	1995 - 1997	$\epsilon$ :0.5 - 1.5%(push/pull)

### 3. Aging tests

Aging condition: (400), (500), 550, 600, 650C; 1000, 3000, 10000, (30000) h

	Property	Test condition	Schedule
Metallurgical tests			1995 -
Mechanical properties	Tensile	RT, 550°C	1995 -
	Charpy Impact	Full curve	1995 -
	Fracture toughness( $K_{IC}$ , $J_{IC}$ )	RT, 100, -30°C	1995 -

4. Weldments (TIG/EB)

*Metallurgical tests*

Test	Schedule	Remarks
Macro/Micro structure	1995	Optical, SEM, TEM microscopy
Hardness	1995	
CCT curve	1995 - 1996	

*Mechanical properties*

Property	Test condition	Schedule	Remarks
Tensile	RT-600°C	1995 - 1996	max: 10000h ε:0.5 - 1.0%(push/pull)
Charpy Impact	Full curve	1995 - 1996	
Fracture toughness(K <sub>IC</sub> , J <sub>IC</sub> )	RT, 100, -30°C	1995 - 1996	
Creep Rapture	500 - 600°C	1996 - 1998	
Fatigue	550°C	1995 - 1997	

*Aging tests*

Aging condition: 550, 600, 650C; 1000, 3000, 10000 h

	Property	Test condition	Schedule
Metallurgical tests			1995 -
Mechanical properties	Tensile	RT, 550°C	1995 -
	Charpy Impact	Full curve	1995 -
	Fracture toughness(K <sub>IC</sub> , J <sub>IC</sub> )	RT, 100, -30°C	1995 -

5. Physical properties (1995 - 1996)

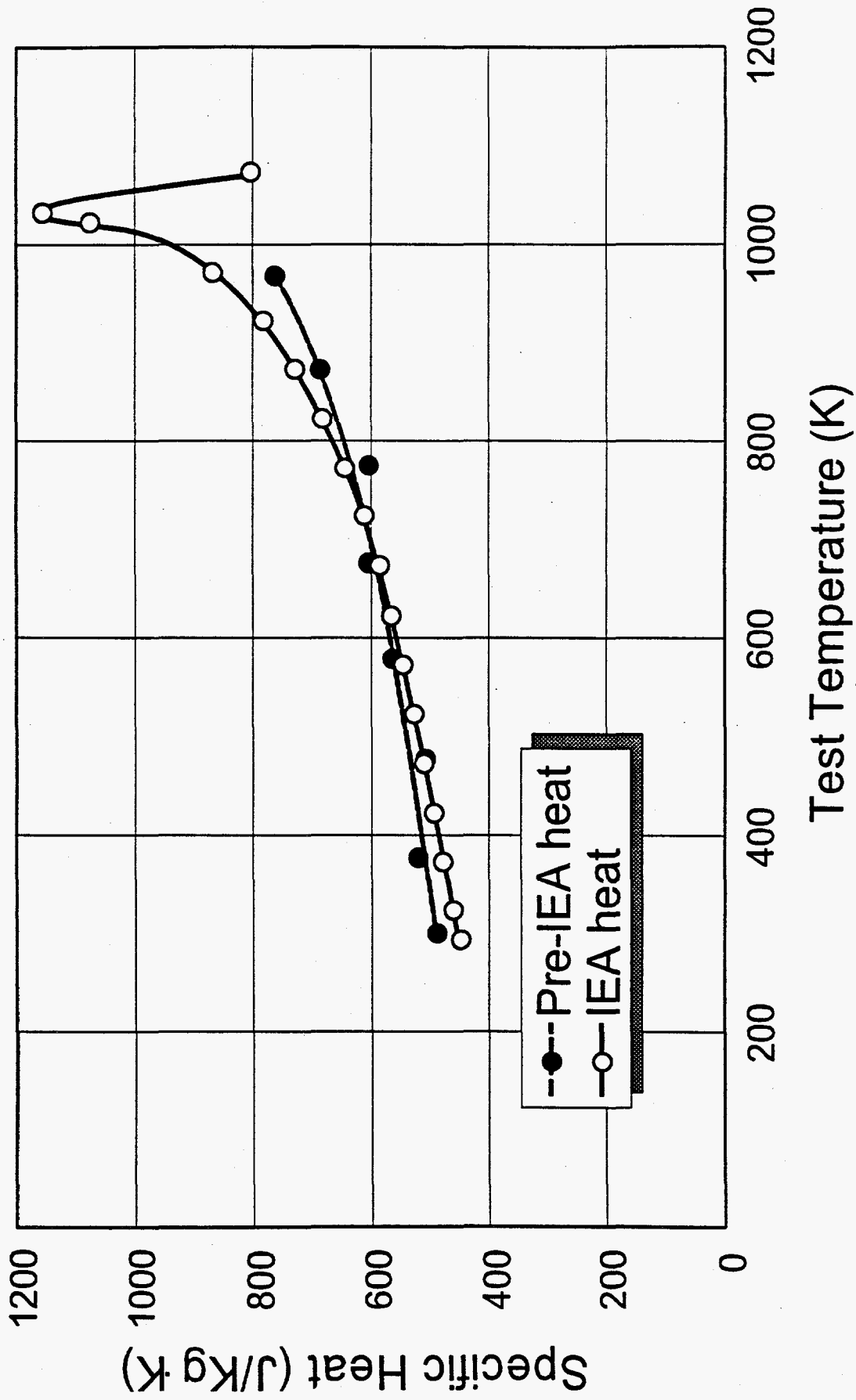
Property	Temperature	Schedule
Density	RT - 700°C	1995
Specific Heat	RT - 700°C	1995
Thermal expansion	RT - 700°C	1995
Thermal conductivity	RT - 700°C	1995
Electrical conductivity	RT - 700°C	1995
Melting point		1995
Young's modules	RT - 700°C	1995
Poisson ratio	RT - 700°C	1995
Modules of rigidity	RT - 700°C	1995
Magnetic hysteresis	RT - 600°C	1995 - 1996

6. Other properties

Property	Measurement	Schedule	Remarks
Vacuum Properties	Released gas measurement	1995 - 1996	
Corrosion Resistance	Corrosion test in high temperature water	1995 - 1996	Corrosion loss SSRT test
Hydrogen Test	Tritium permeability	1995 - 1996	Hydrogen solubility after aging
	Hydrogen cracking	1995 - 1996	SSRT test

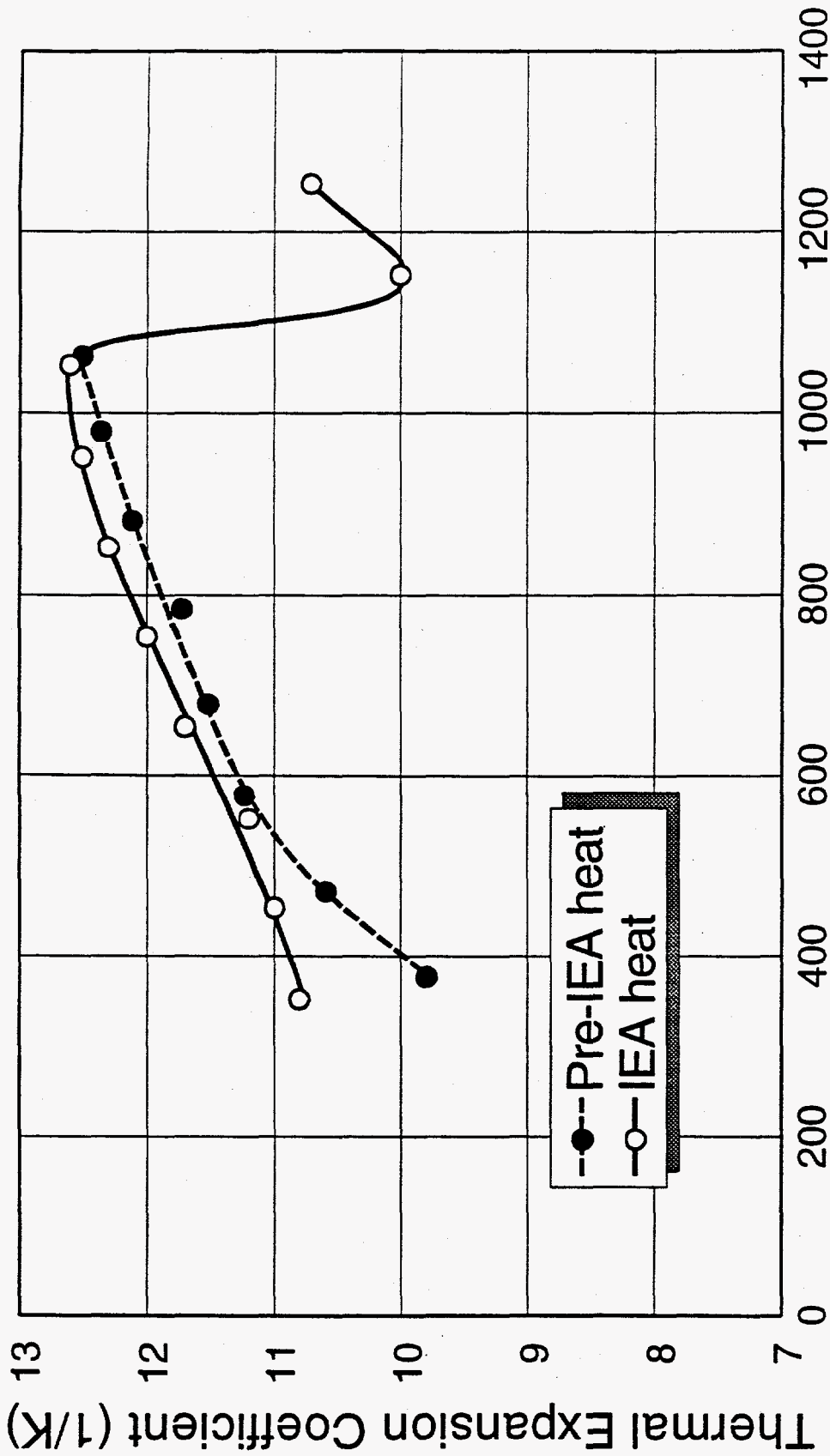


# Specific Heat of F82H



# Thermal Expansion Coefficient of F82H

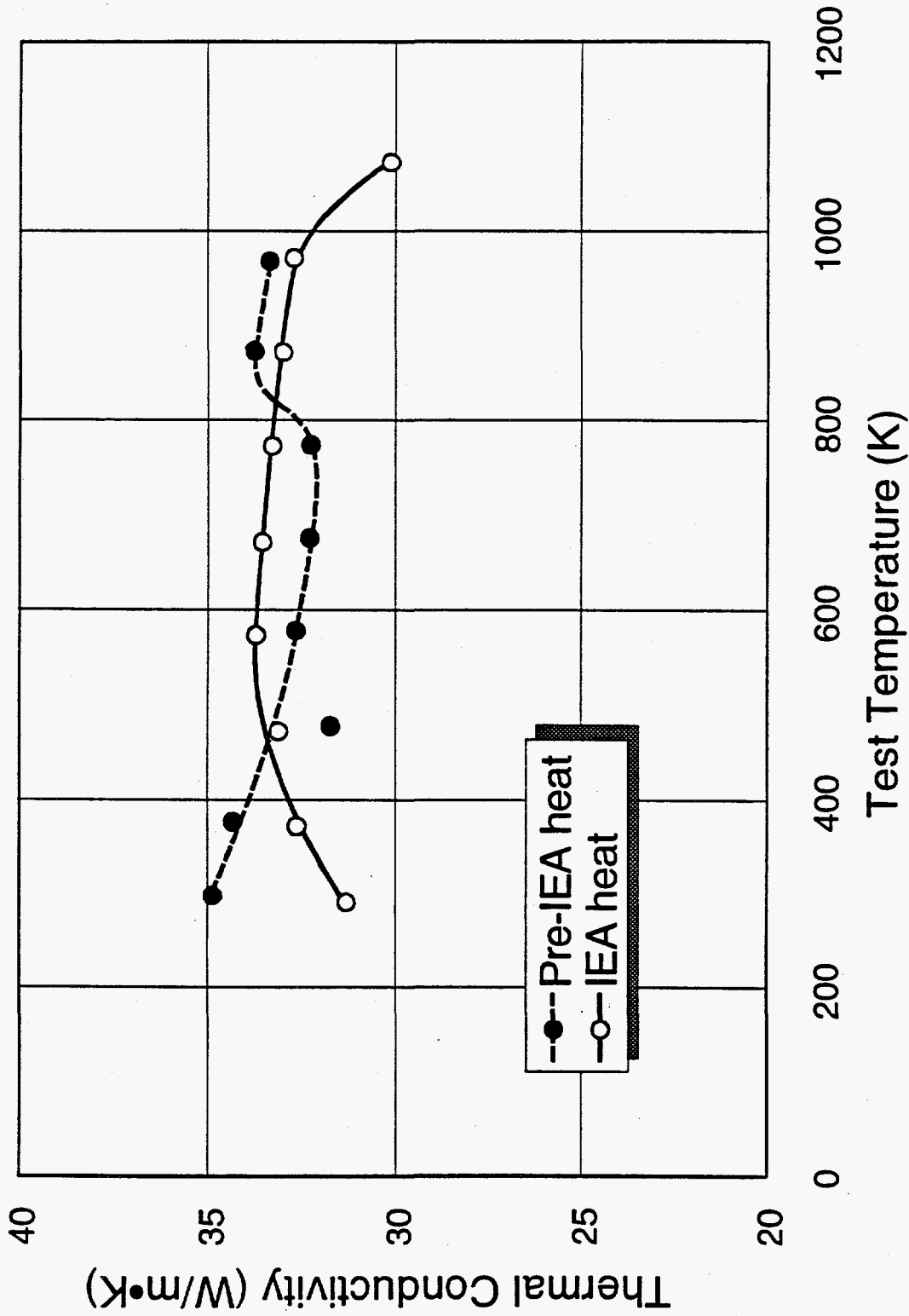
$\times 10^{-6}$



Test Temperature (K)

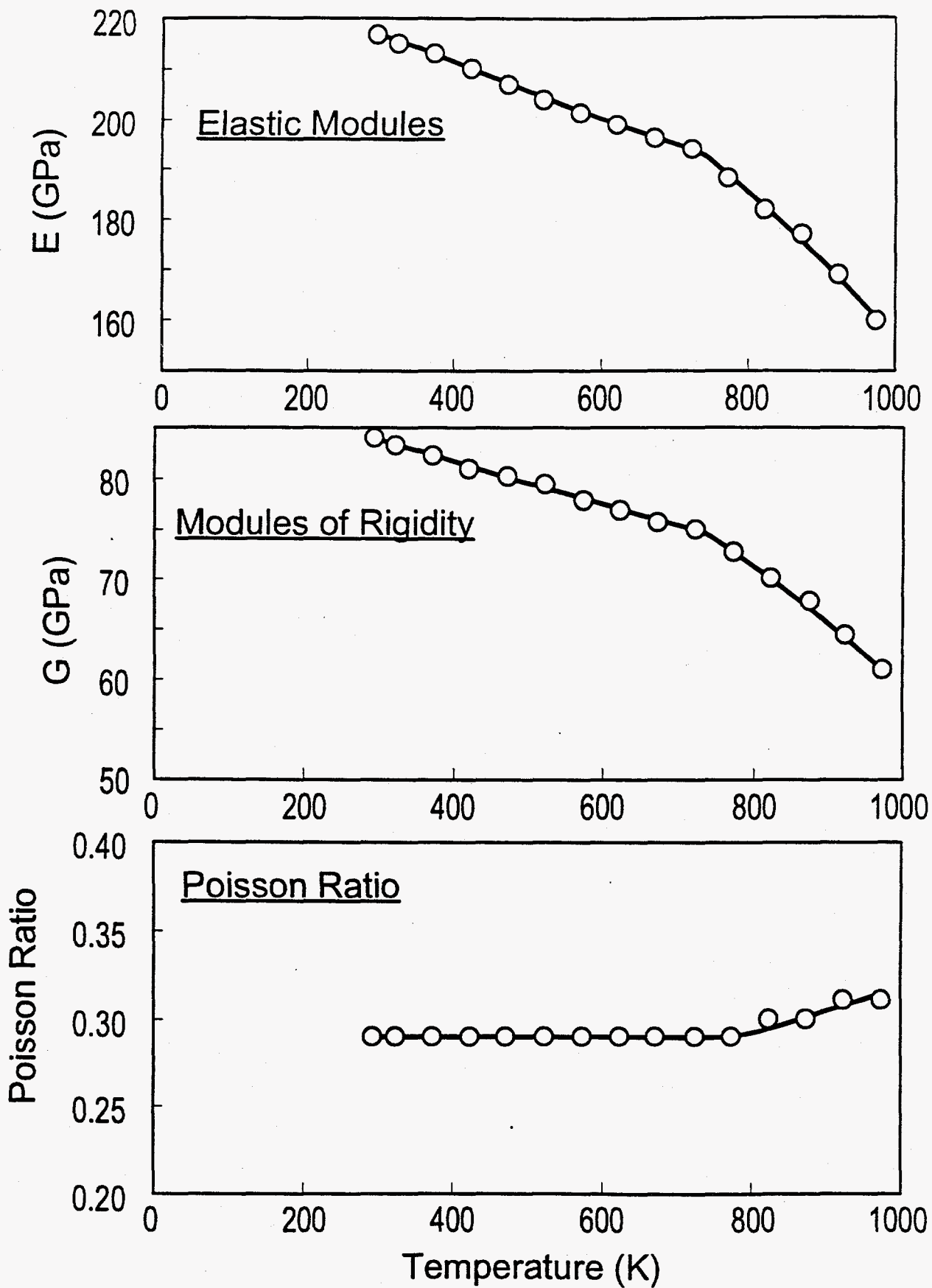
[Ref]: FRM Semi-annual DOE/ER-0313/20 (to be published)

# Thermal Conductivity of F82H



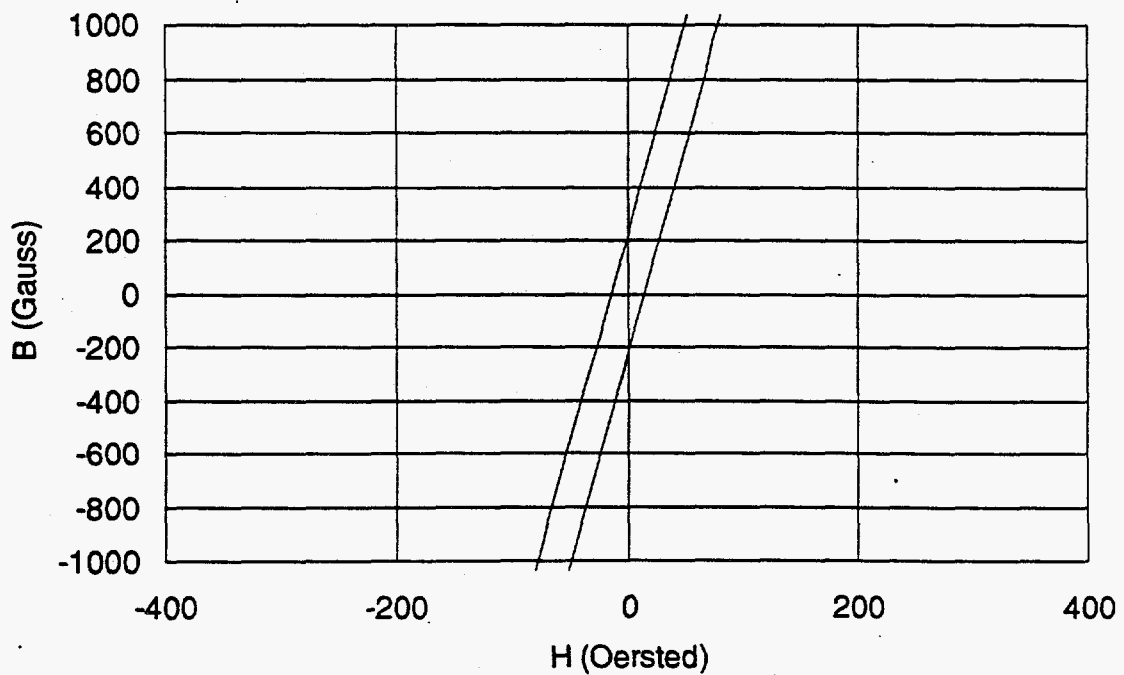
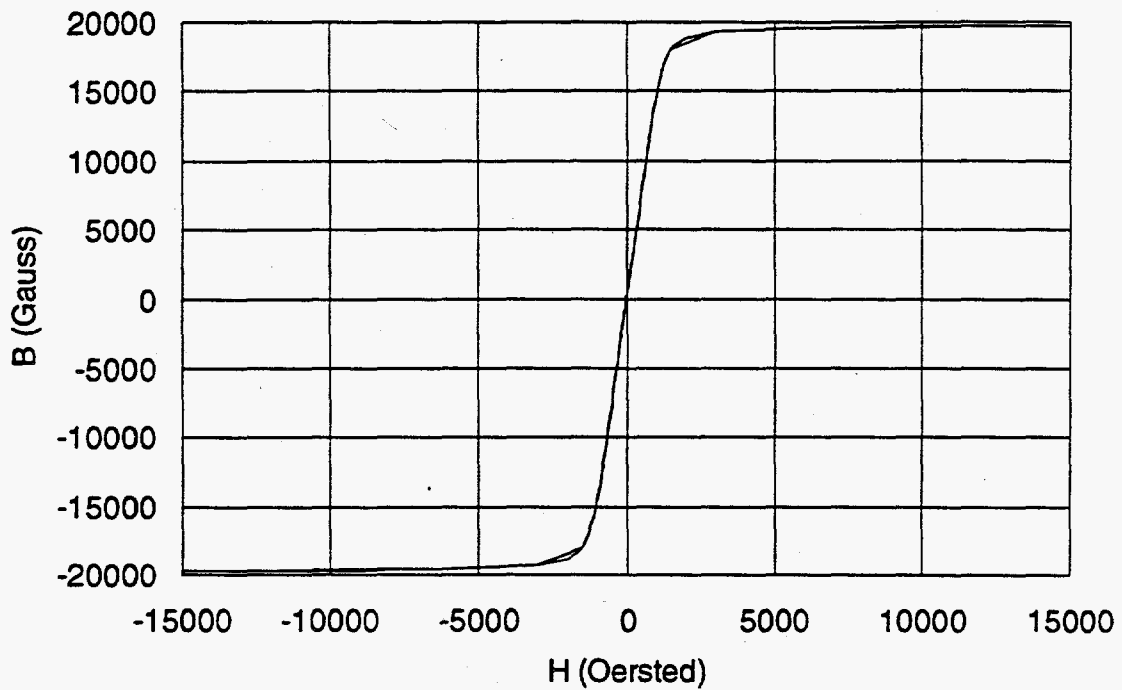
[Ref]: FRM Semi-annual DOE/ER-0313/20 (to be published)

# Physical Properties of F82H IEA Heat



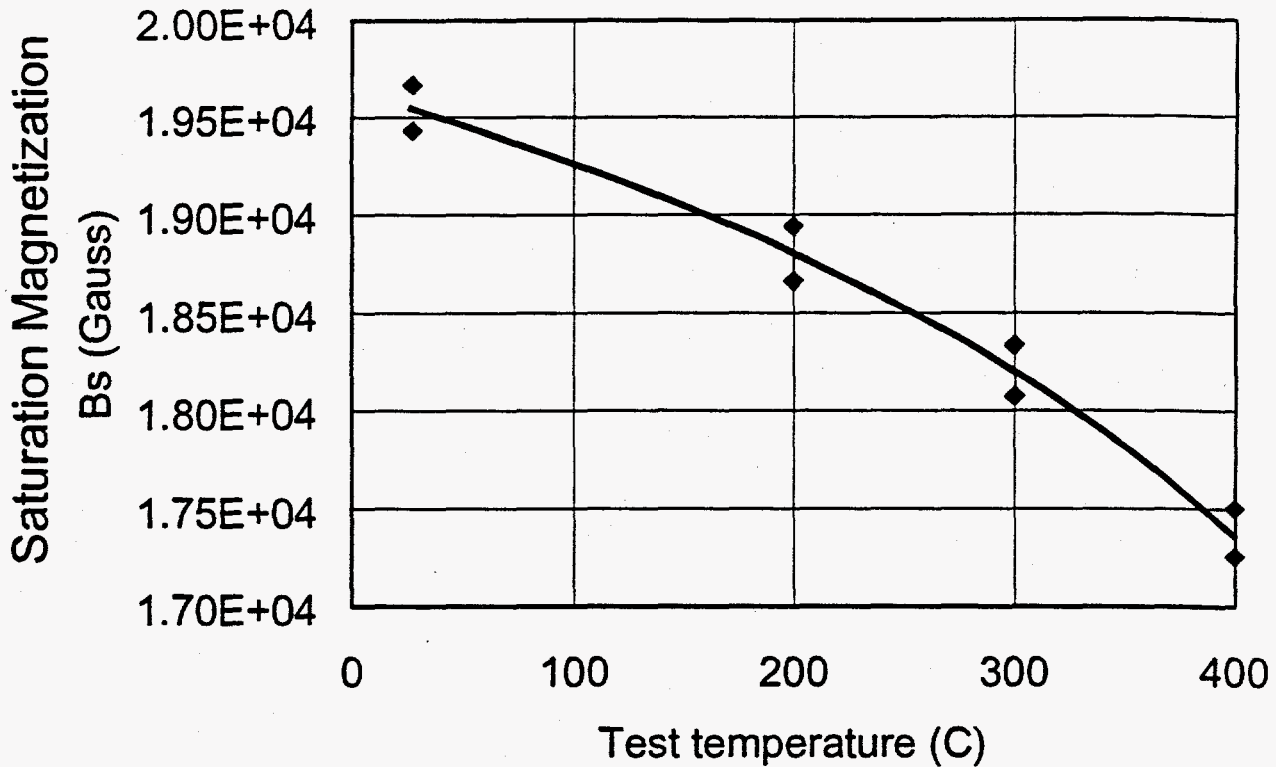
# Hysteresis Curve of F82H (RT)

F82H (RT)

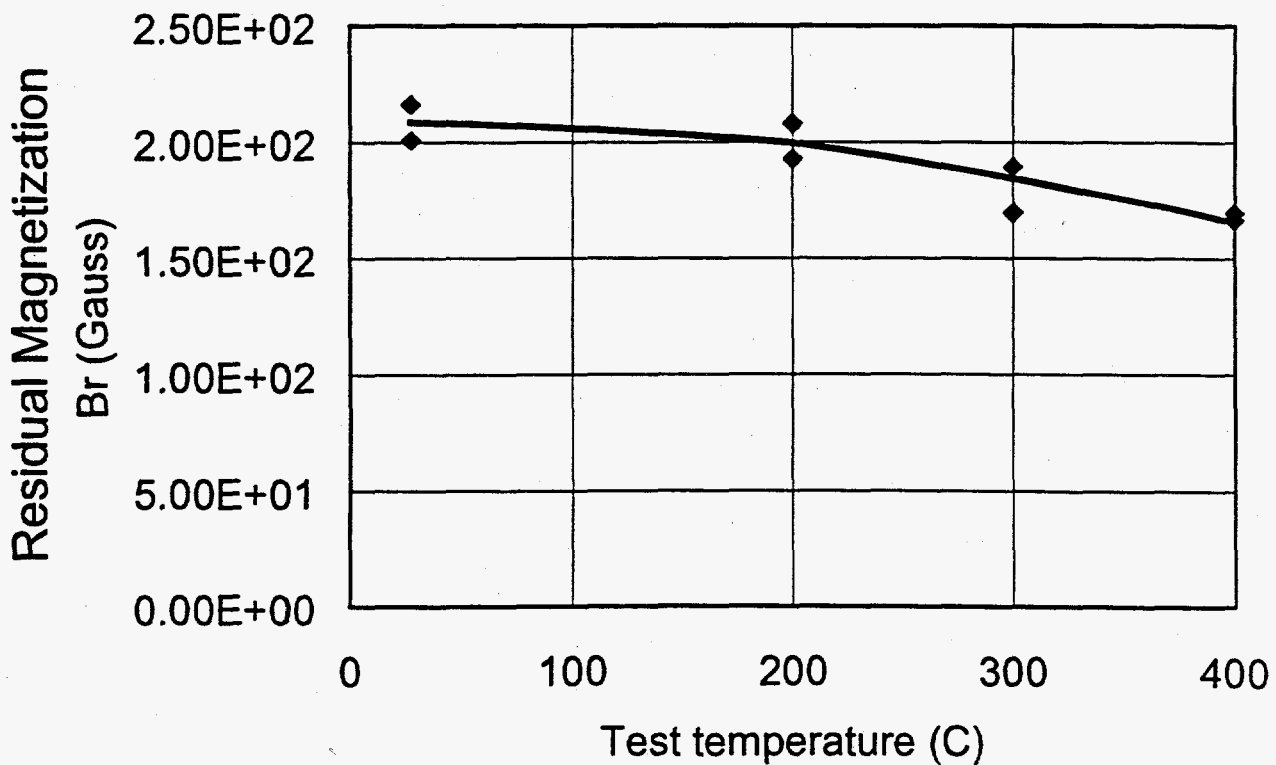


# Magnetic Properties of F82H (IEA heat)

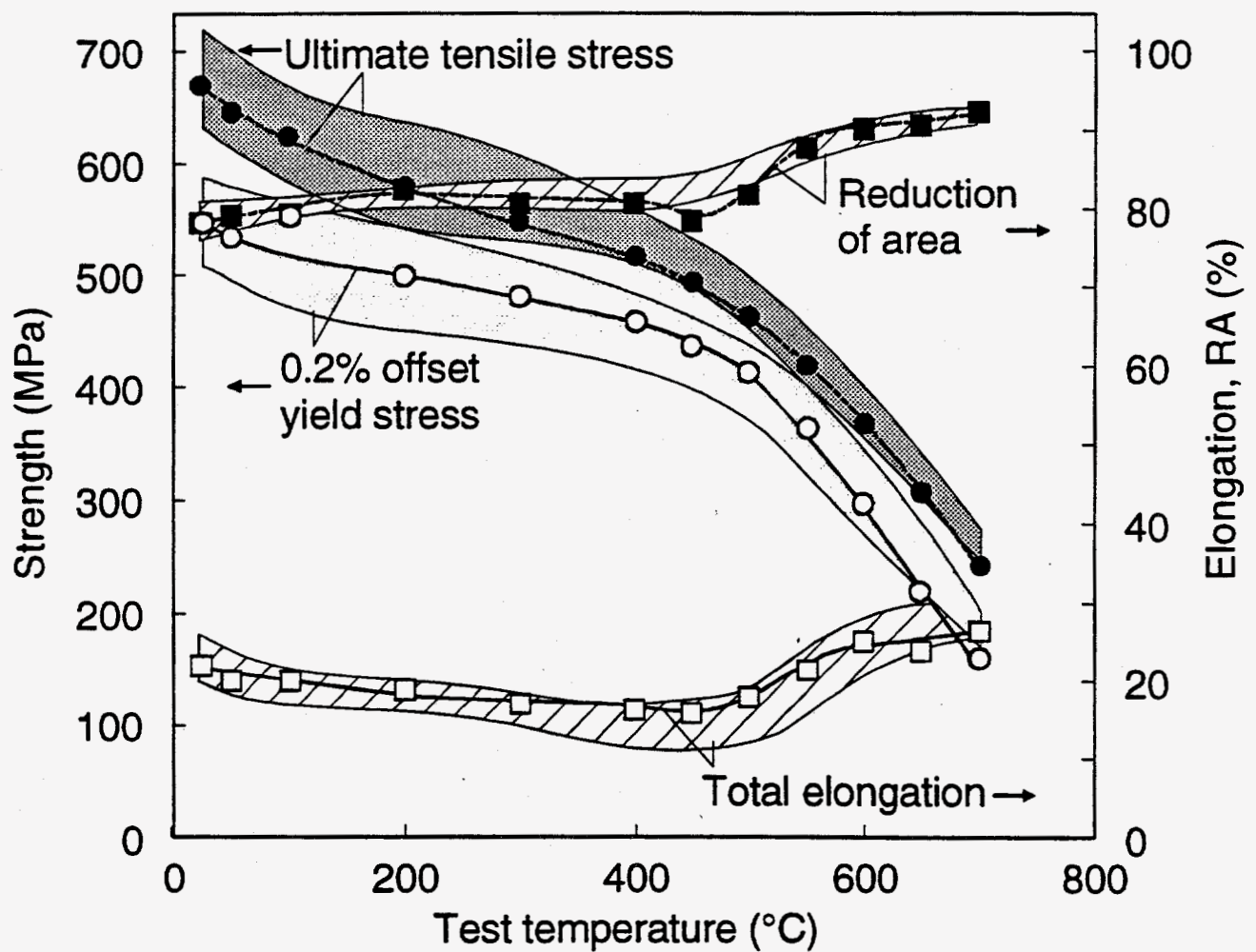
F82H Magnetization Curve Measurement  
(Vibration Sample Magnetometer)



F82H Magnetization Curve Measurement  
(Vibration Sample Magnetometer)

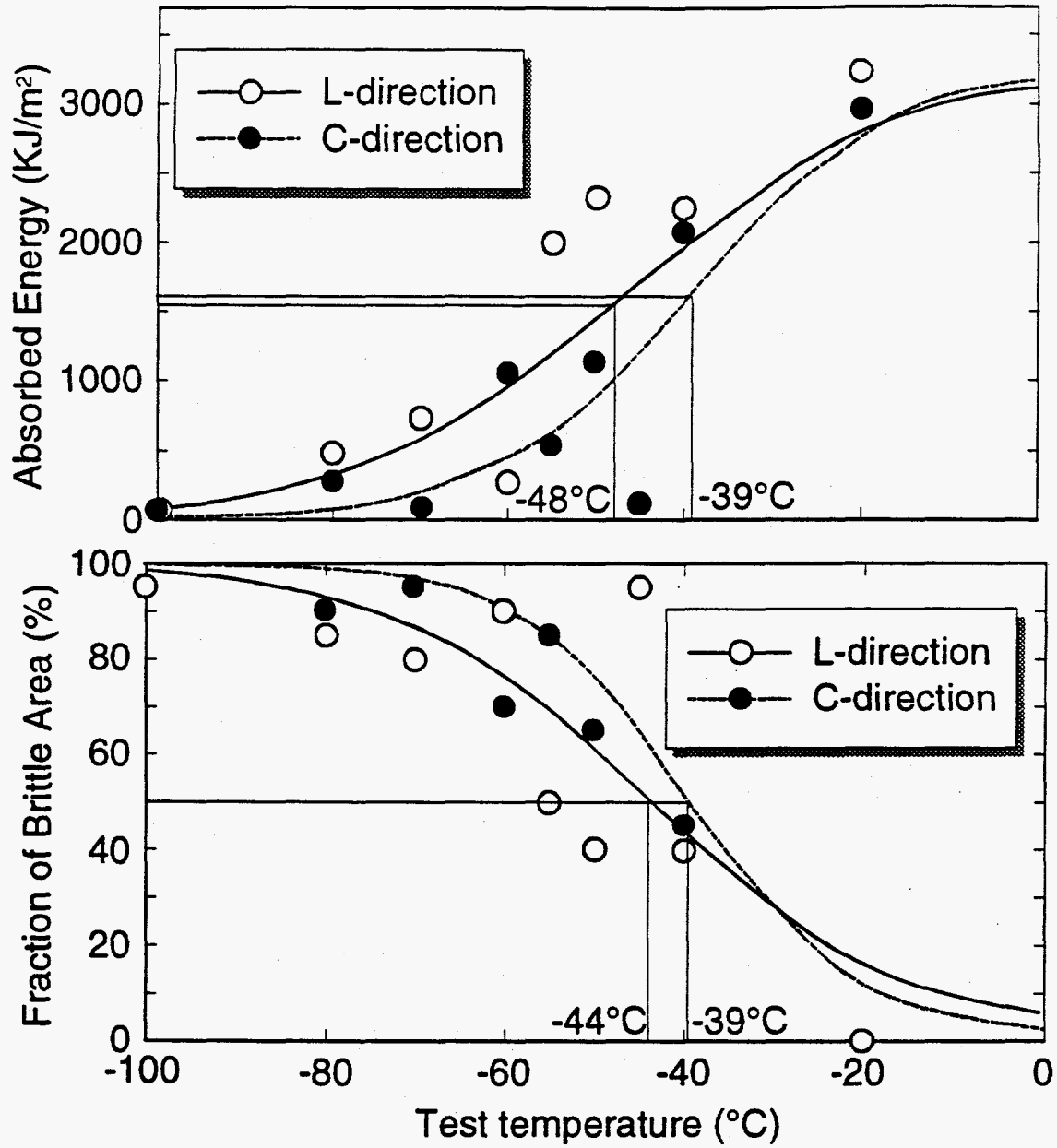


## Tensile Properties of F82H IEA Heat



[Ref]: FRMSemi-annual DOE/ER-0313/20 (to be published)

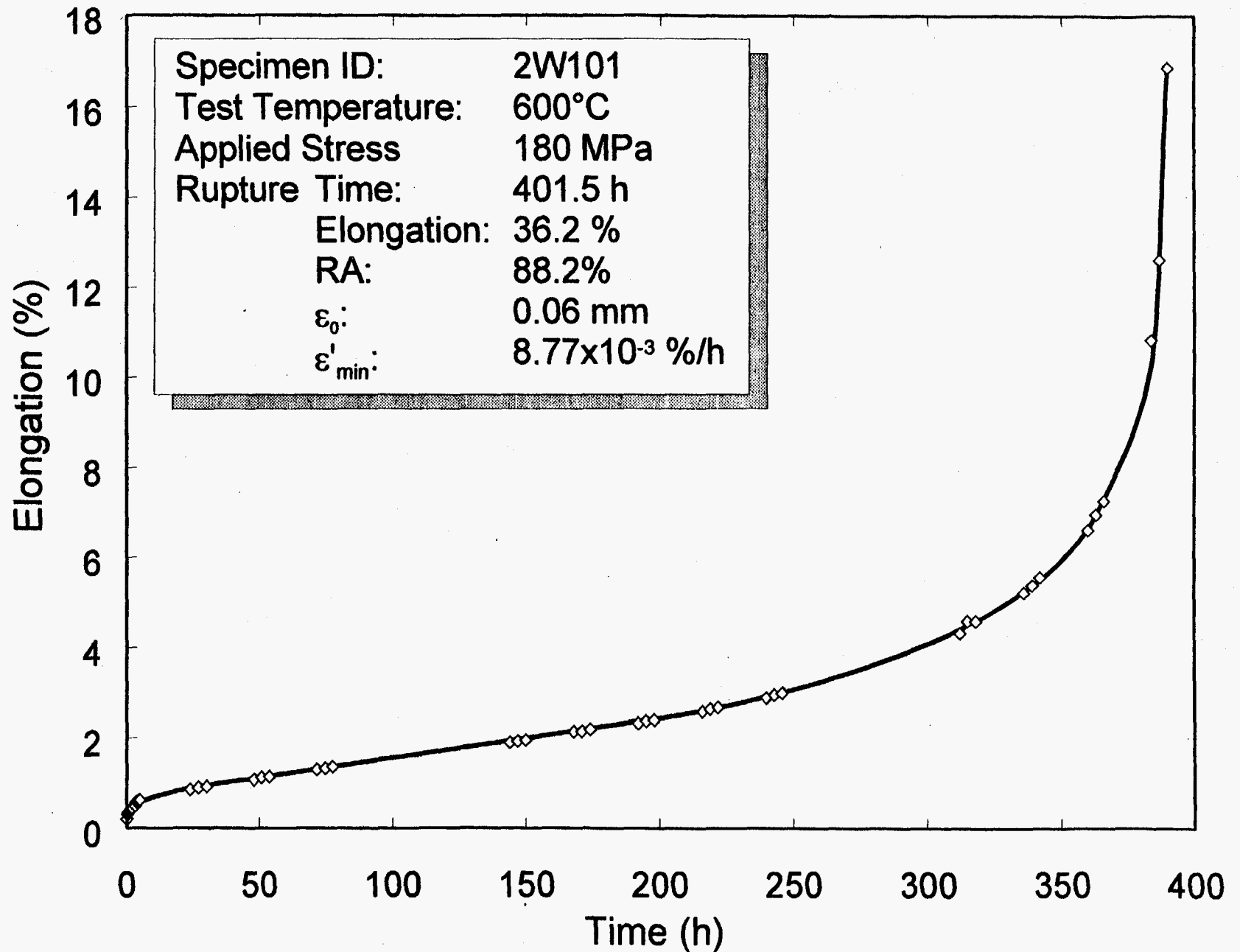
# Charpy Impact Test Results of F82H IEA Heat



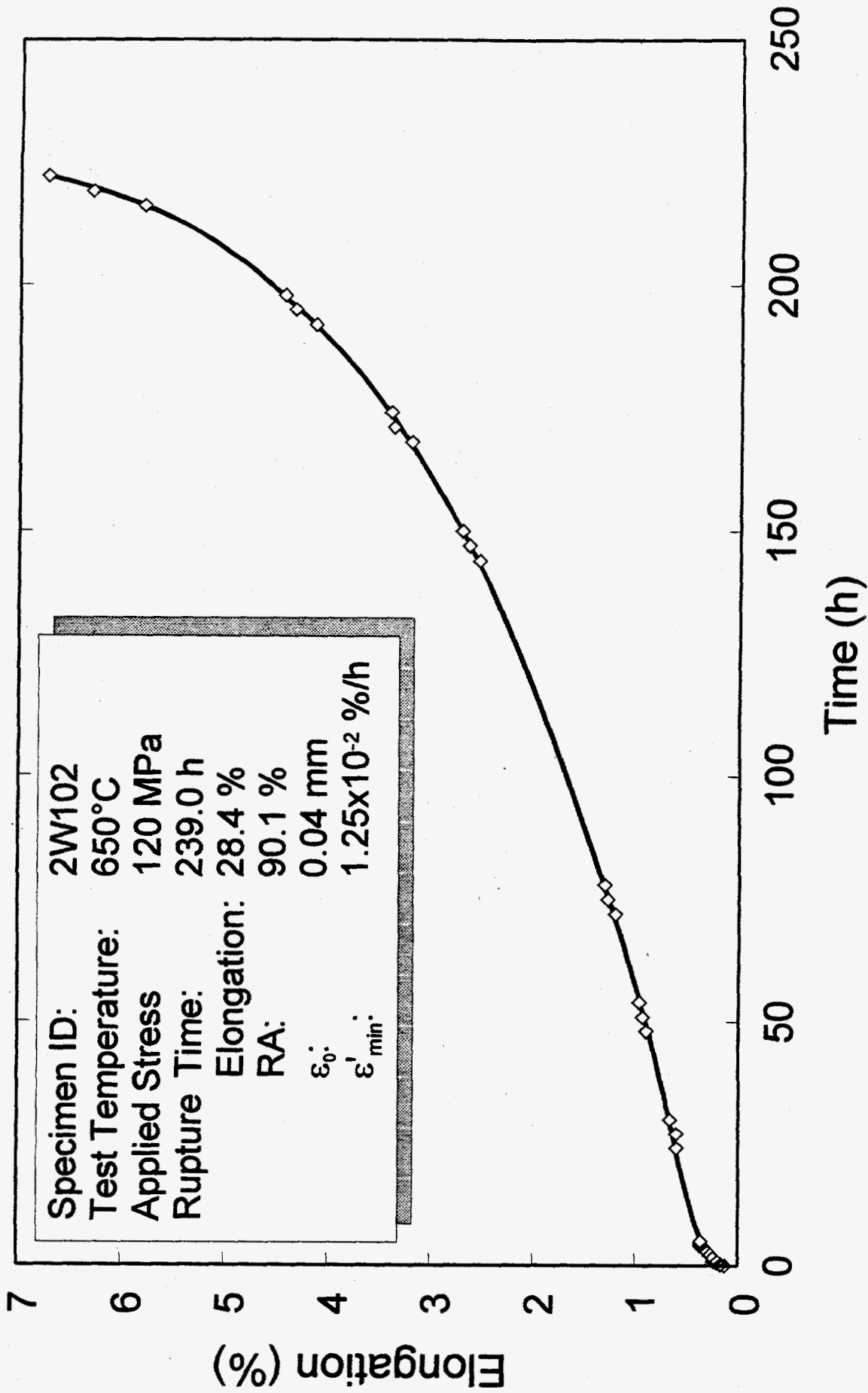
[Ref]: FRMSemi-annual DOE/ER-0313/20 (to be published)



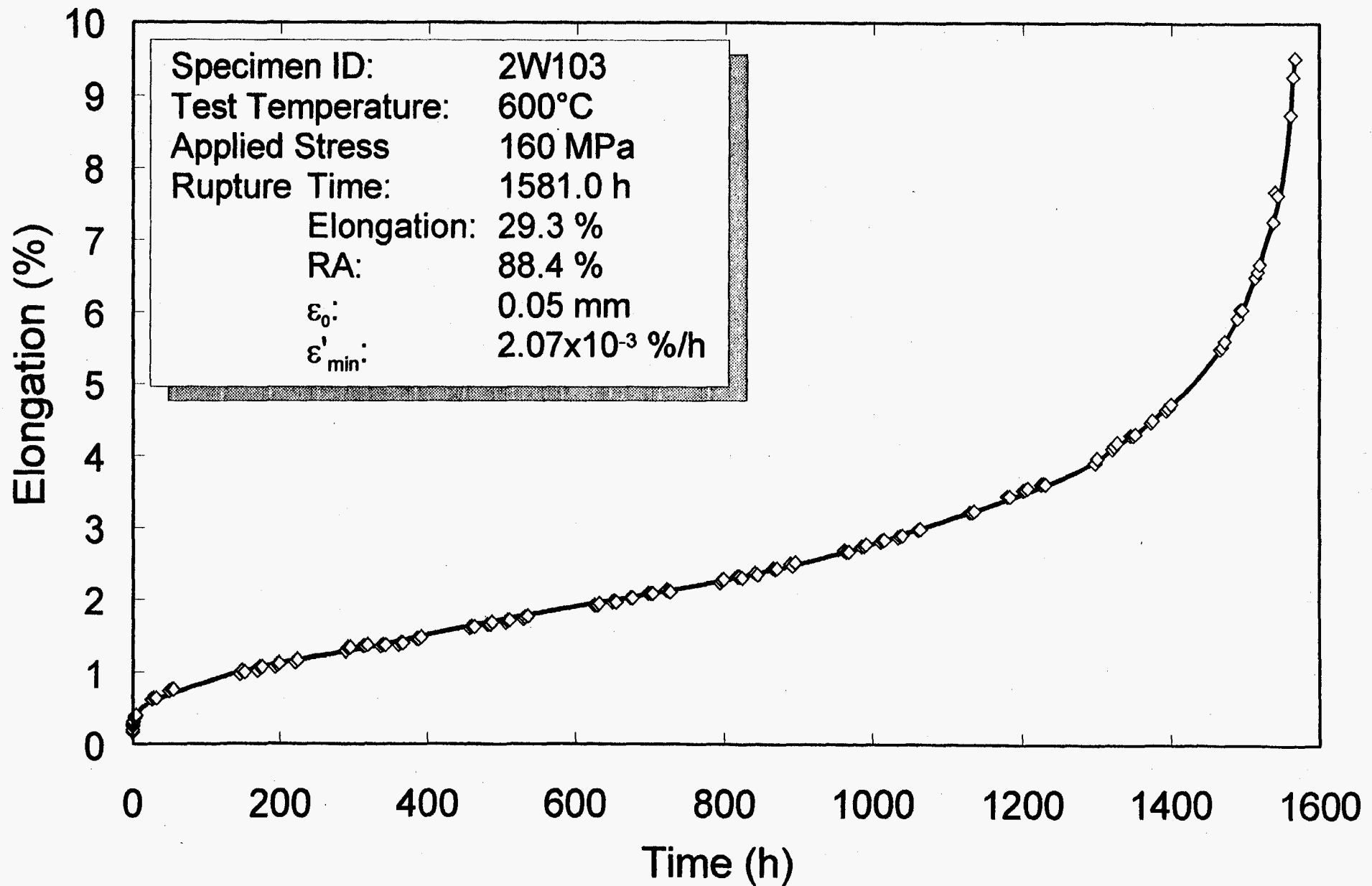
## Creep Rupture Test Result of F82H IEA Heat (2W101)



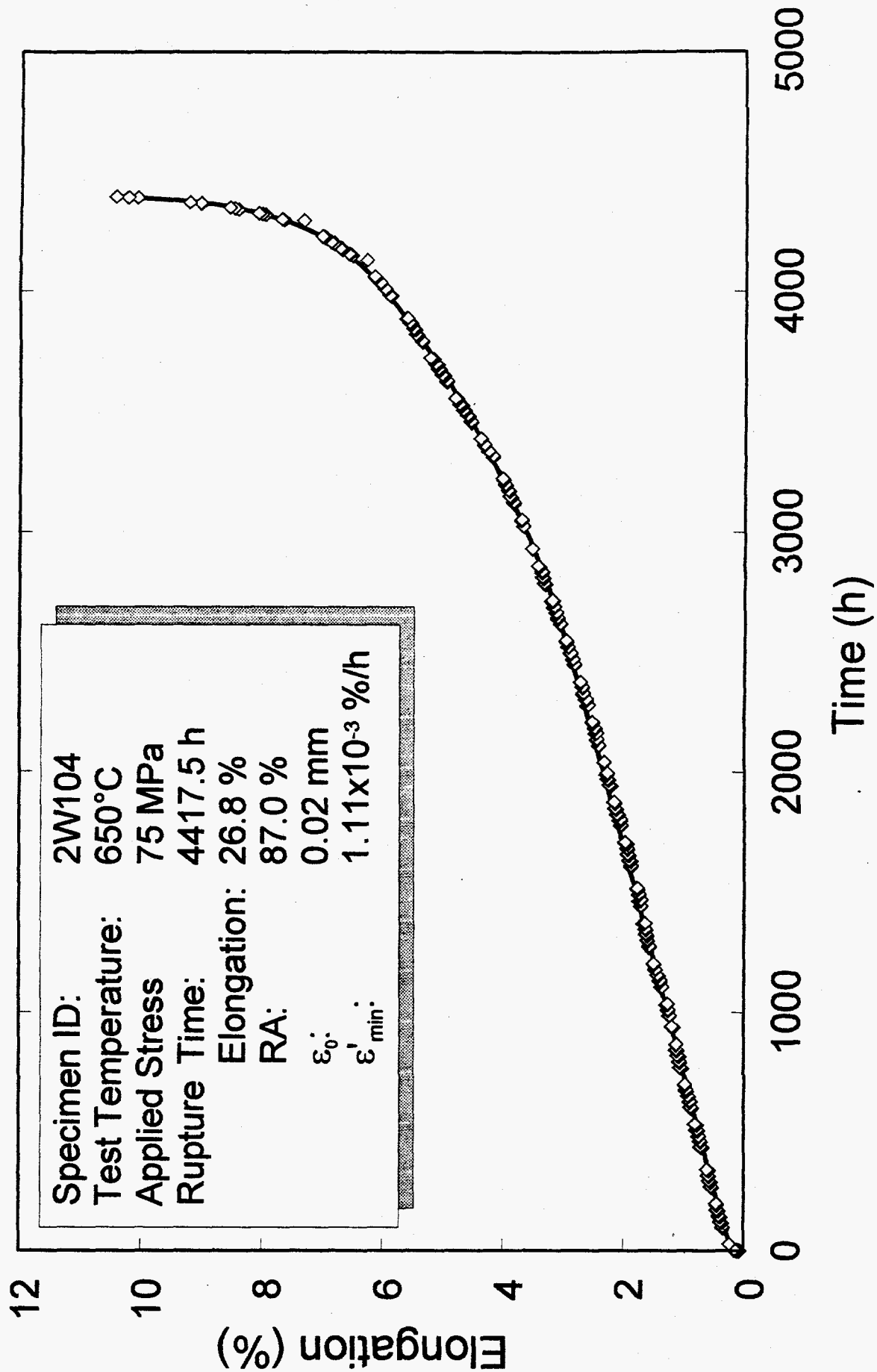
# Creep Rupture Test Result of F82H IEA Heat (2W102)



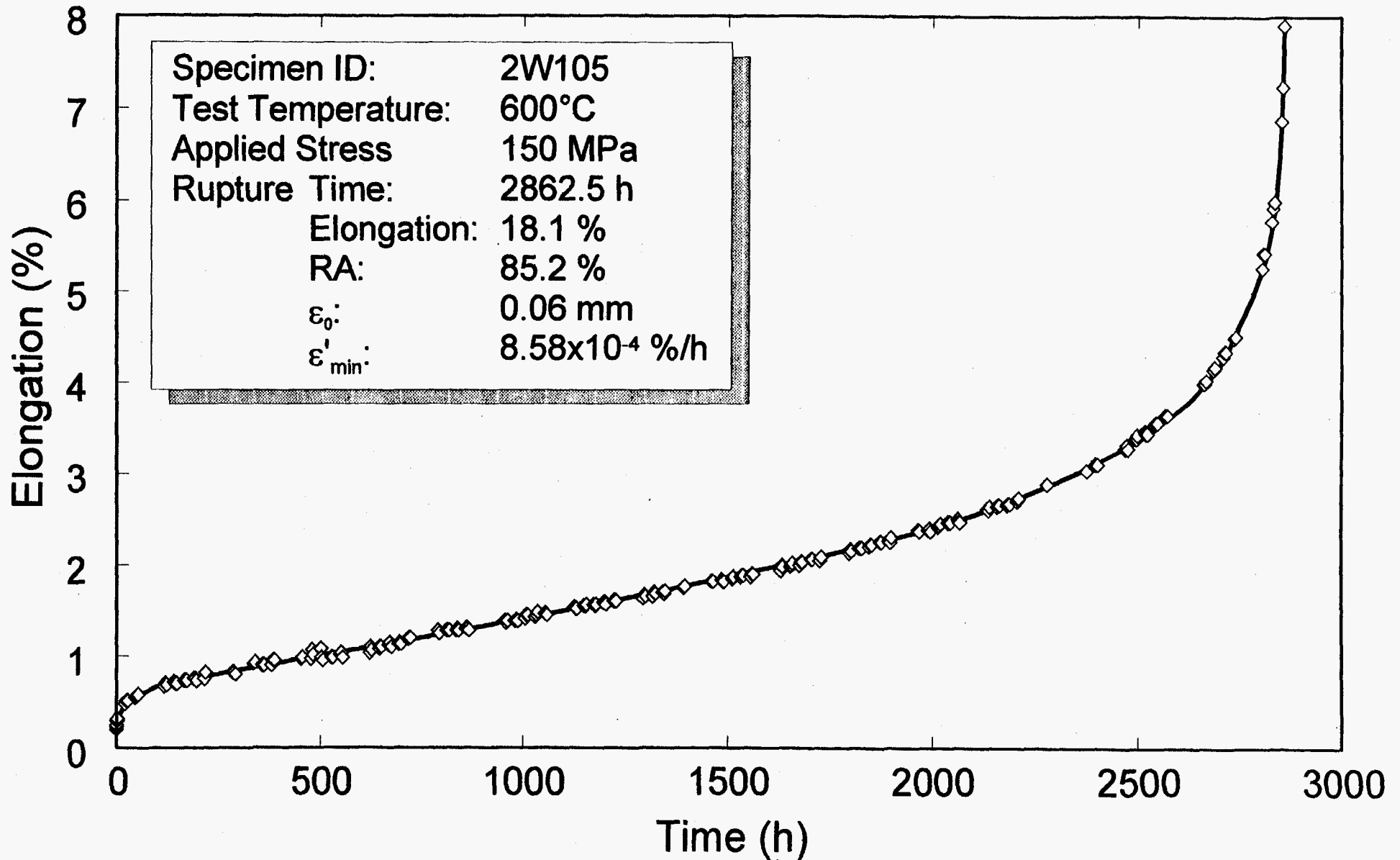
## Creep Rupture Test Result of F82H IEA Heat (2W103)



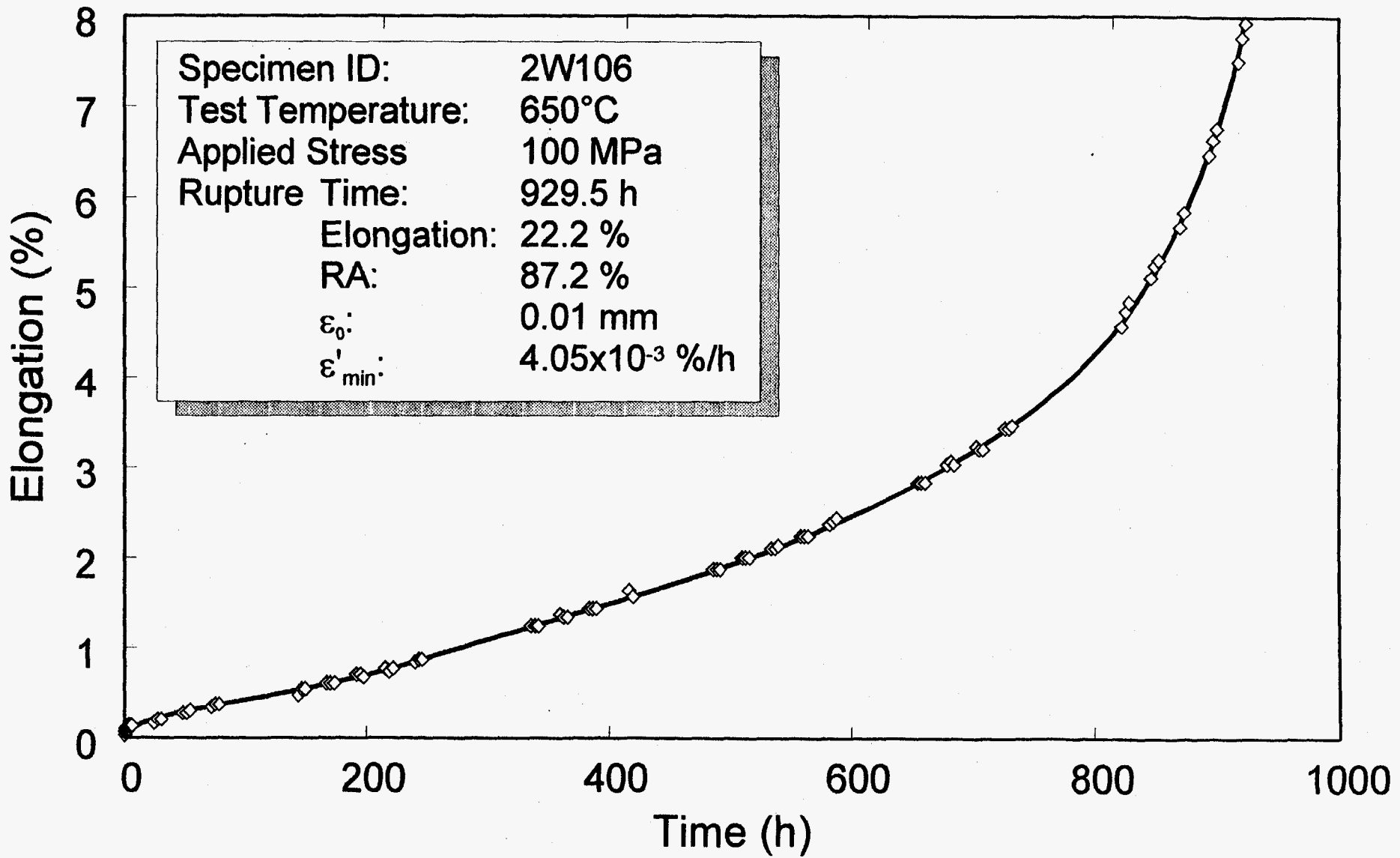
# Creep Rupture Test Result of F82H IEA Heat (2W104)



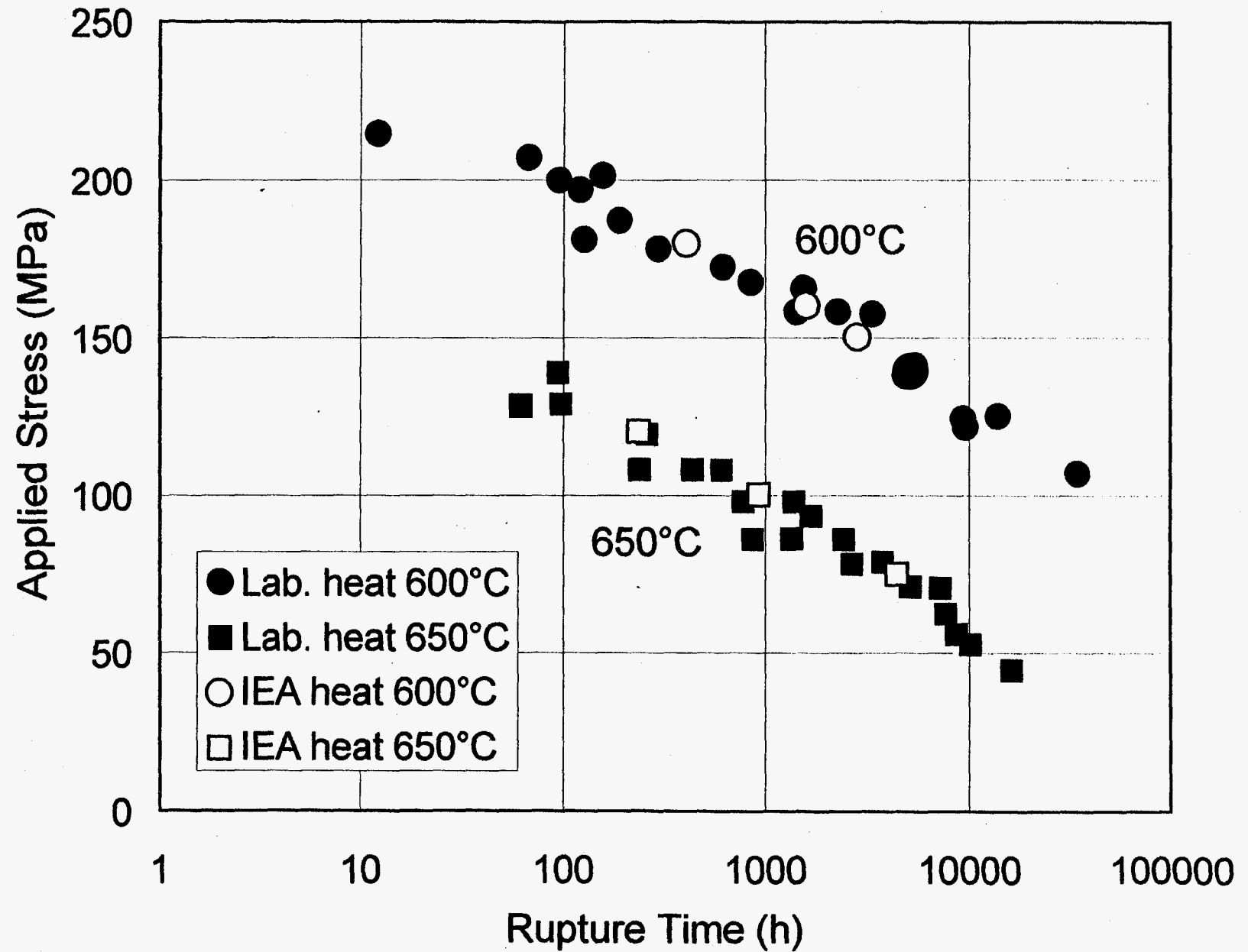
## Creep Rupture Test Result of F82H IEA Heat (2W105)



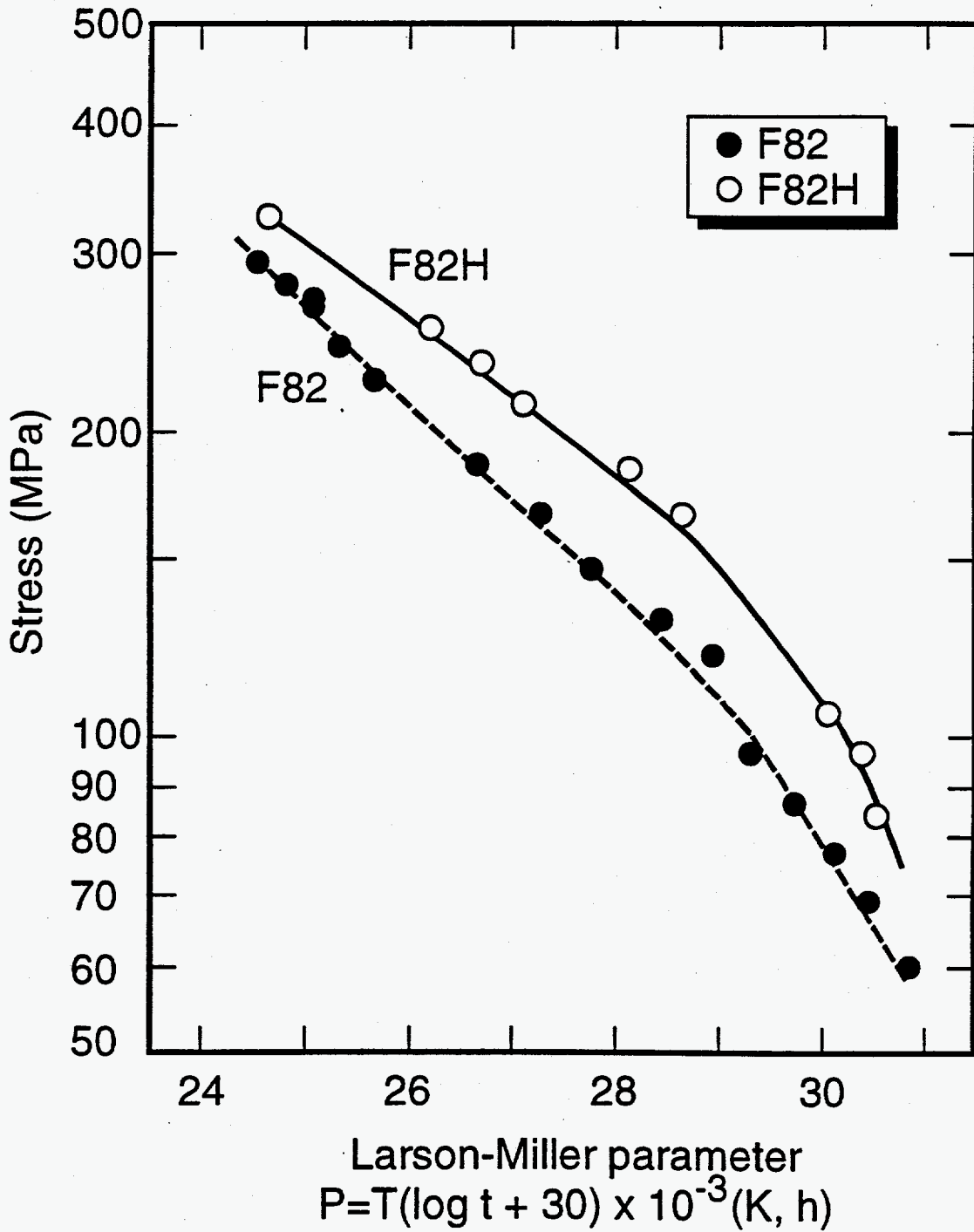
## Creep Rupture Test Result of F82H IEA Heat (2W106)



## Creep Rupture Test Results of F82H



# Larson-Miller Plot of Creep Rapture Data





STATUS OF AGING TEST

Test Items \ Aging		Base Metal	1000h				3000h			
			500°C	550°C	600°C	650°C	500°C	550°C	600°C	650°C
Microstructure	x100	○	△	△	△	△	△	△	△	○
	x400	○	○	△	△	△	○	△	△	○
Extrusion Analysis		○	—	—	○	○	○	○	○	○
X-ray Analysis		○	—	—	○	○	○	○	○	○
SEM (EDX, etc.)		○	—	—	○	○	○	○	○	○
TEM		△	—	—	—	—	—	—	—	○
Hardness		○	△	△	△	△	△	△	△	○
Tensile (RT, 550°C)		○	○	○	○	○	○	○	○	○
Charpy (full curve)		○	○	○	○	○	○	○	○	○
Fracture Toughness (J-R)		○	—	—	—	—	—	—	—	—

○: finished, △: in progress, —: not scheduled

10000, 30000, 100000h (500, 550, 600, 650°C) in aging process

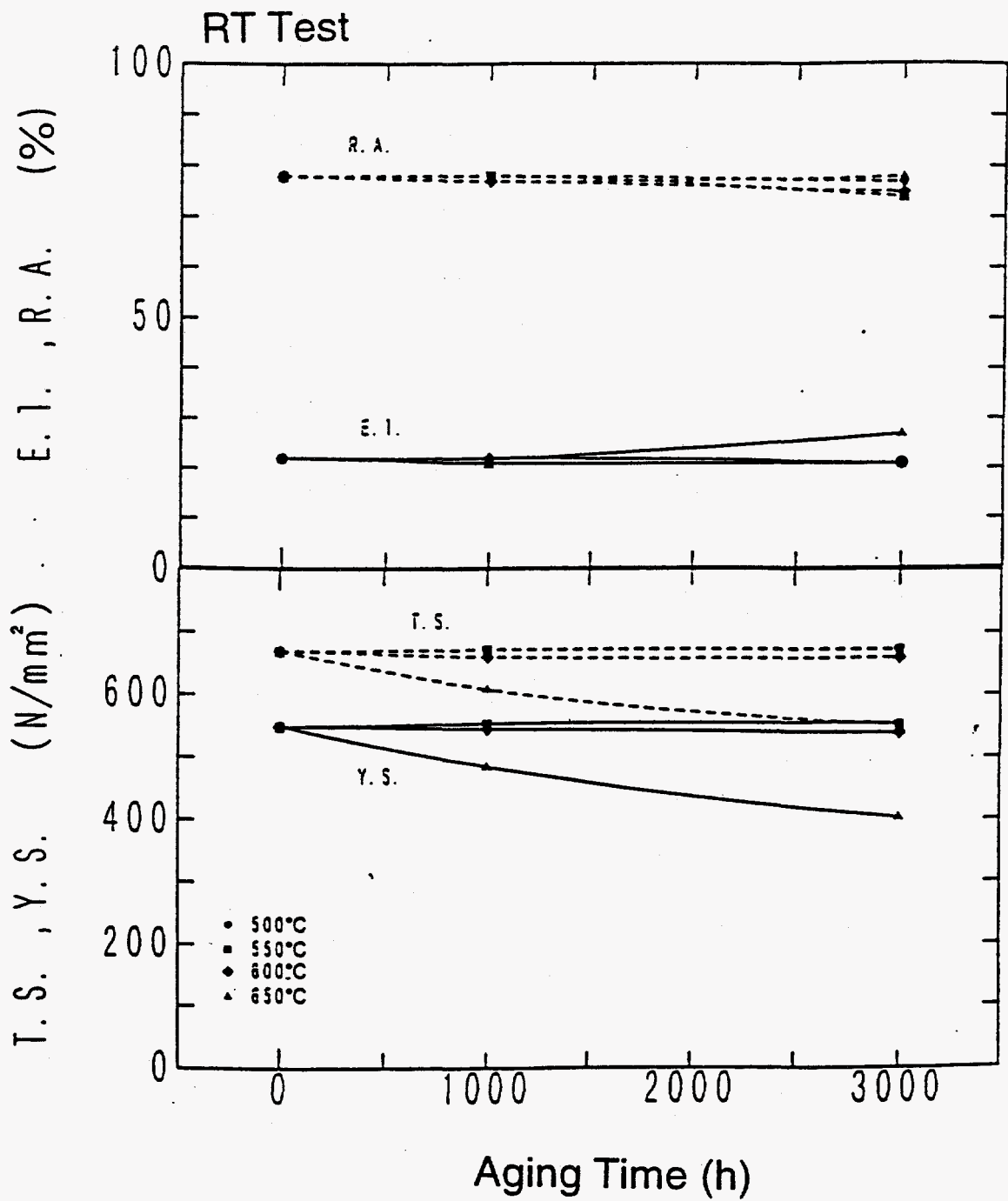
## RESULTS

### Hardness Measurement

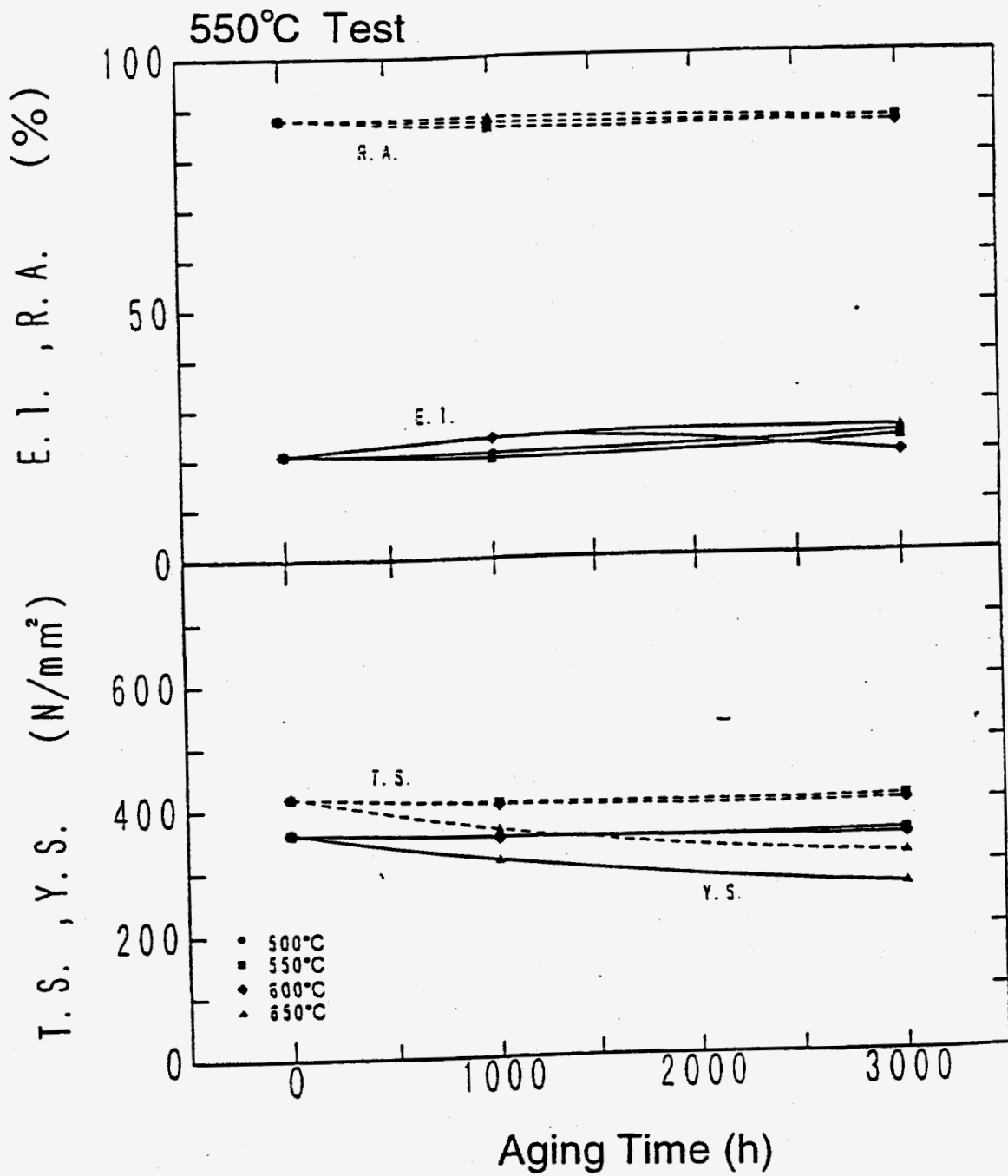
Aging		Plate	ID	Thickness mm	Hardness Hv10
Temp	Time				
Base Metal		RB801-5	5-16	7.5	216
		KG819-2	2W-23	15	219
		KG820-2	42W-18	25	213
650°C	3000h	KG819-2	2W-15	15	190

### Extraction Residue Analysis

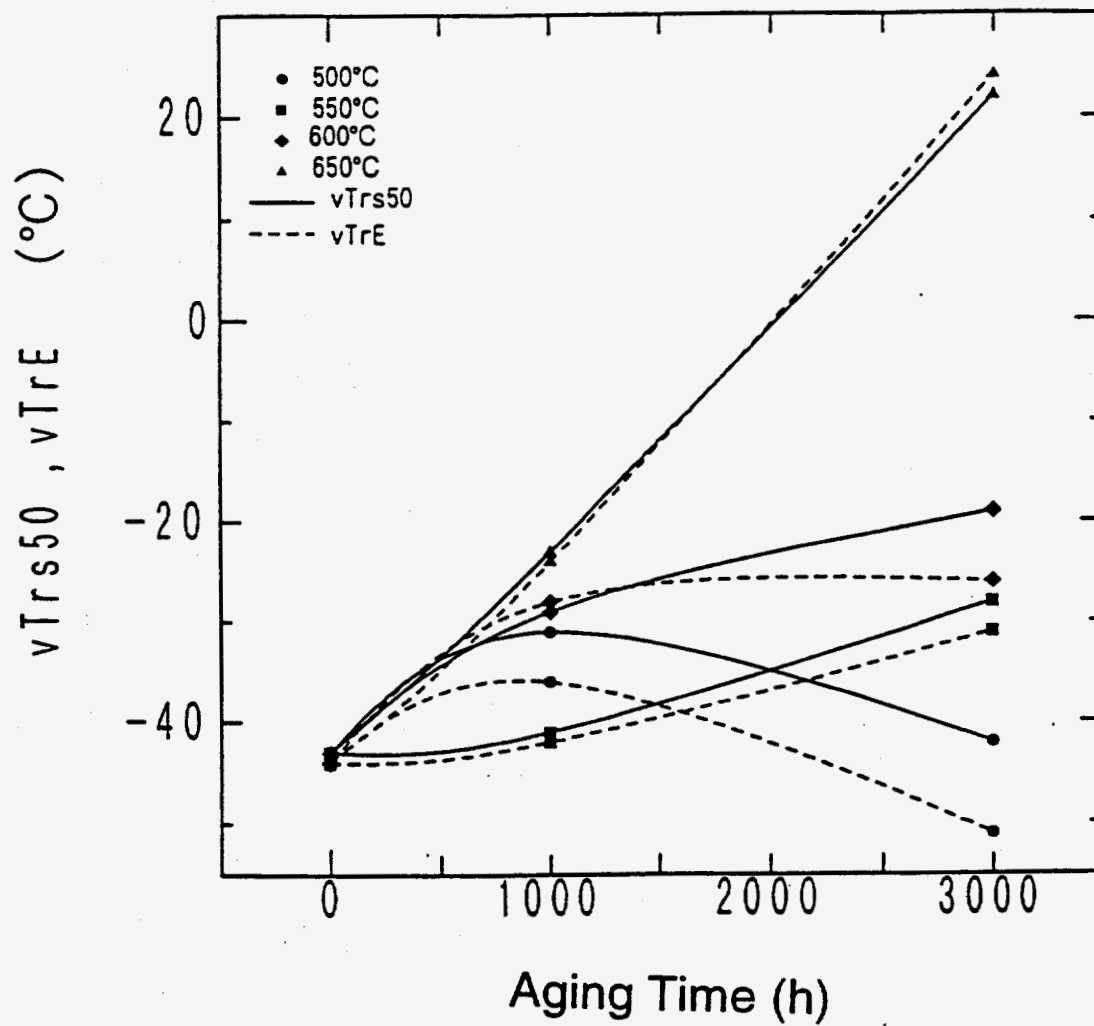
Aging		ID	Element						
Temp	Time		W	Cr	Fe	V	Mn	Ta	Si
Base Metal		2W-10	0.20	0.81	0.42	0.06	0.003	0.01	<0.01
600°C	1000h	2W-12	0.26	0.89	0.42	0.06	0.003	0.01	<0.01
650°C		2W-13	0.26	0.95	0.37	0.07	0.004	0.01	<0.01
500°C	3000h	2W-14	0.21	0.84	0.40	0.06	0.003	0.01	<0.01
550°C		2W-14	0.28	0.89	0.44	0.06	0.004	0.01	<0.01
600°C		2W-15	0.41	0.96	0.48	0.07	0.004	0.01	<0.01
650°C		2W-15	0.42	1.01	0.43	0.07	0.004	0.01	<0.01



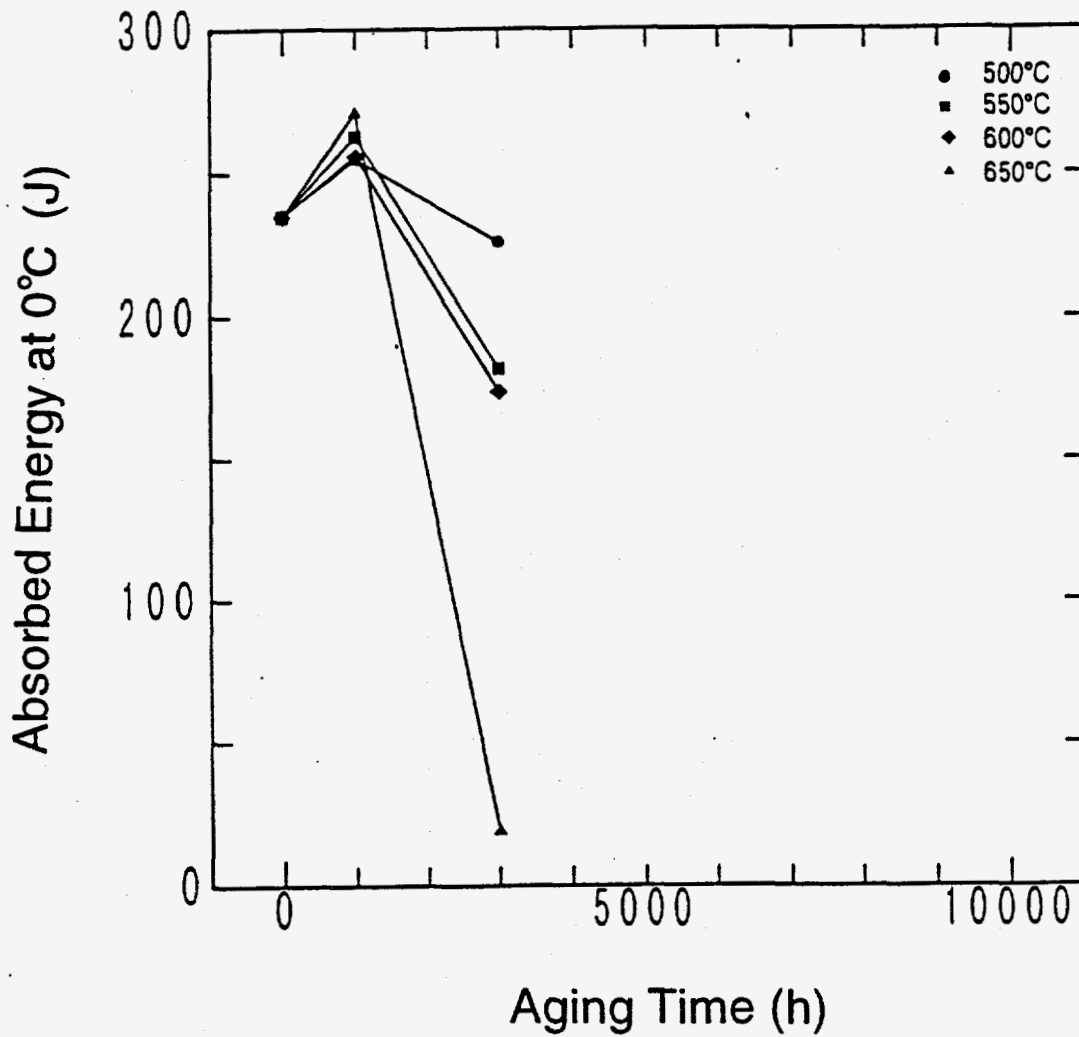
Tensile Test Result after Aging



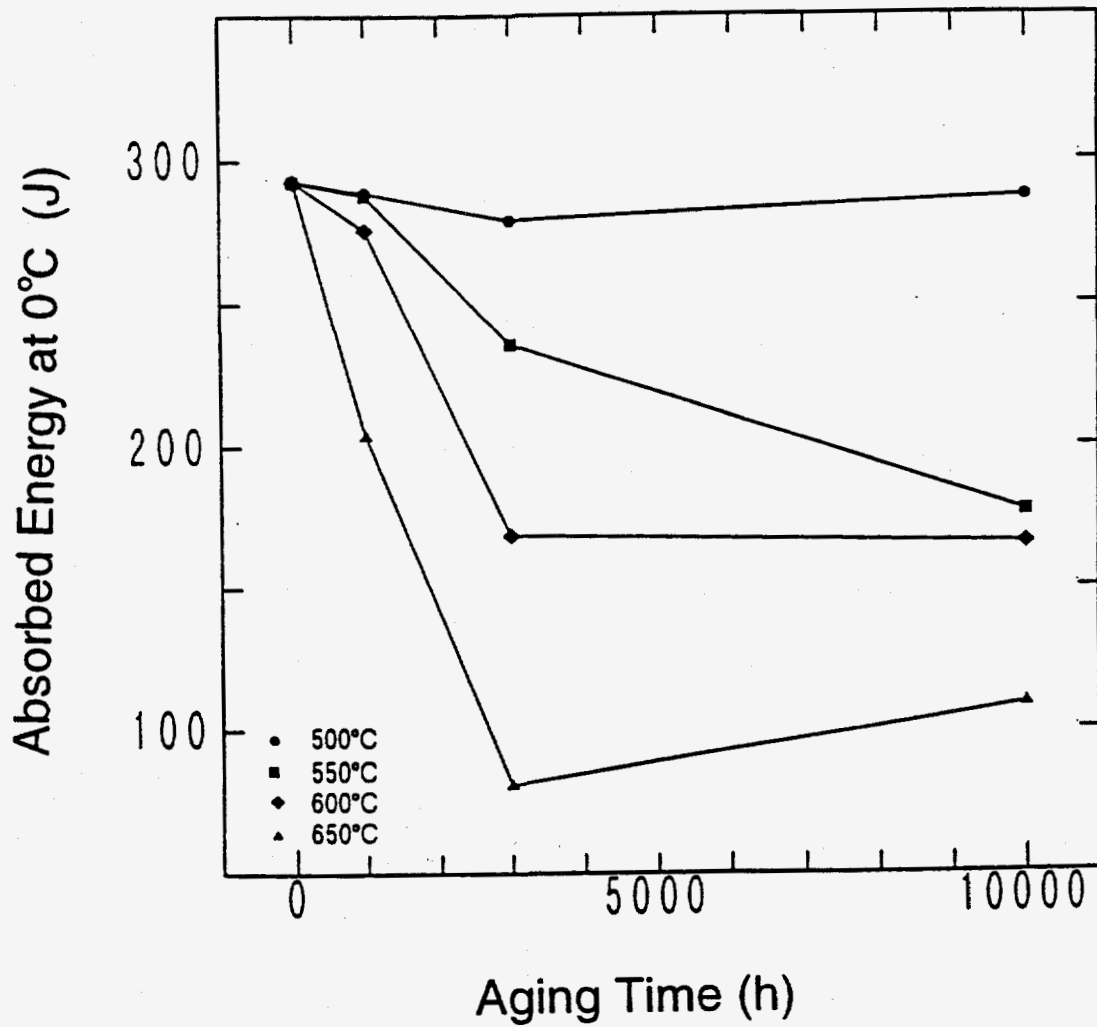
Tensile Test Result after Aging



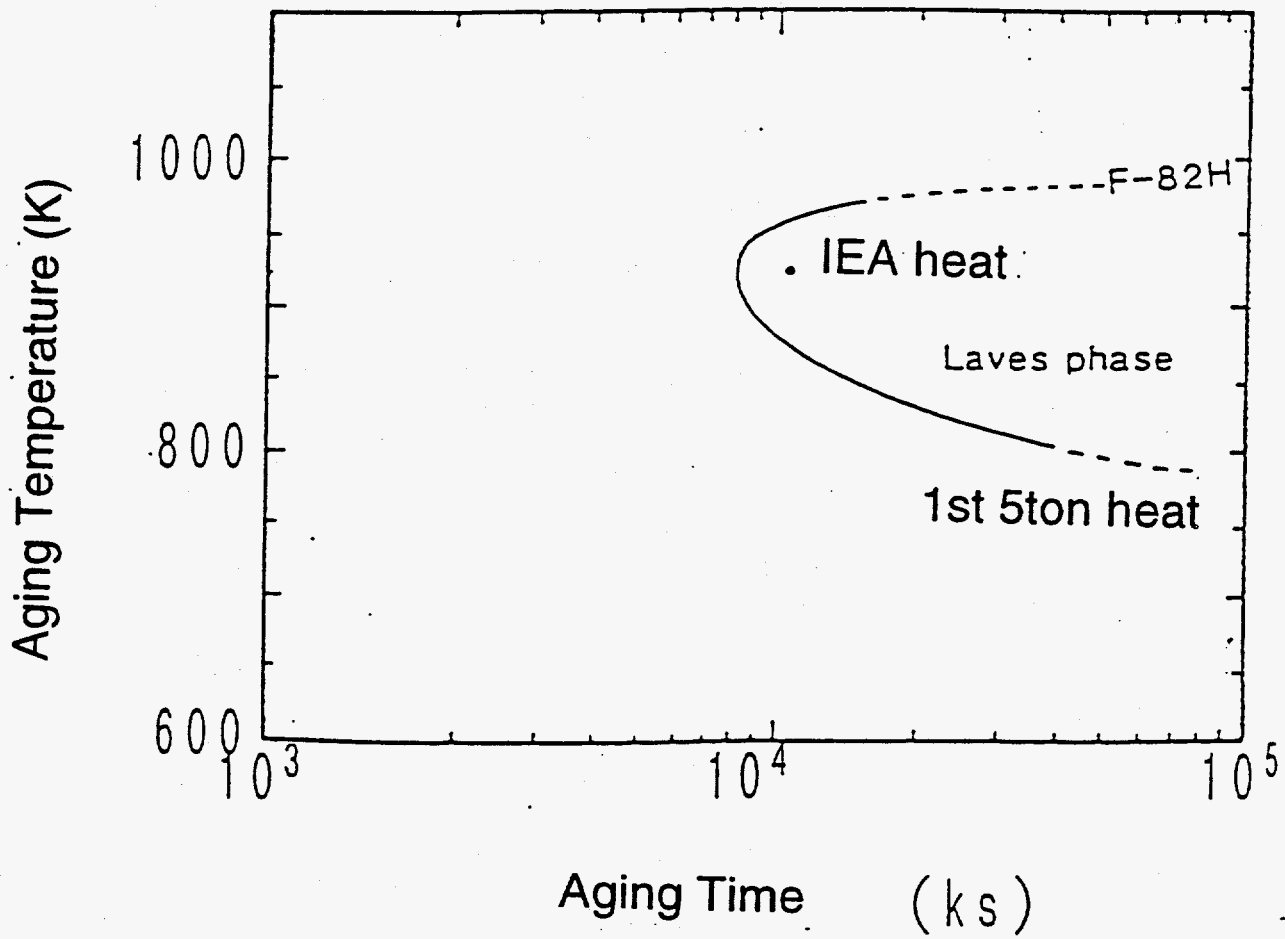
DBTT after Aging



Absorbed Energy at 0°C after Aging (IEA 5ton heat)



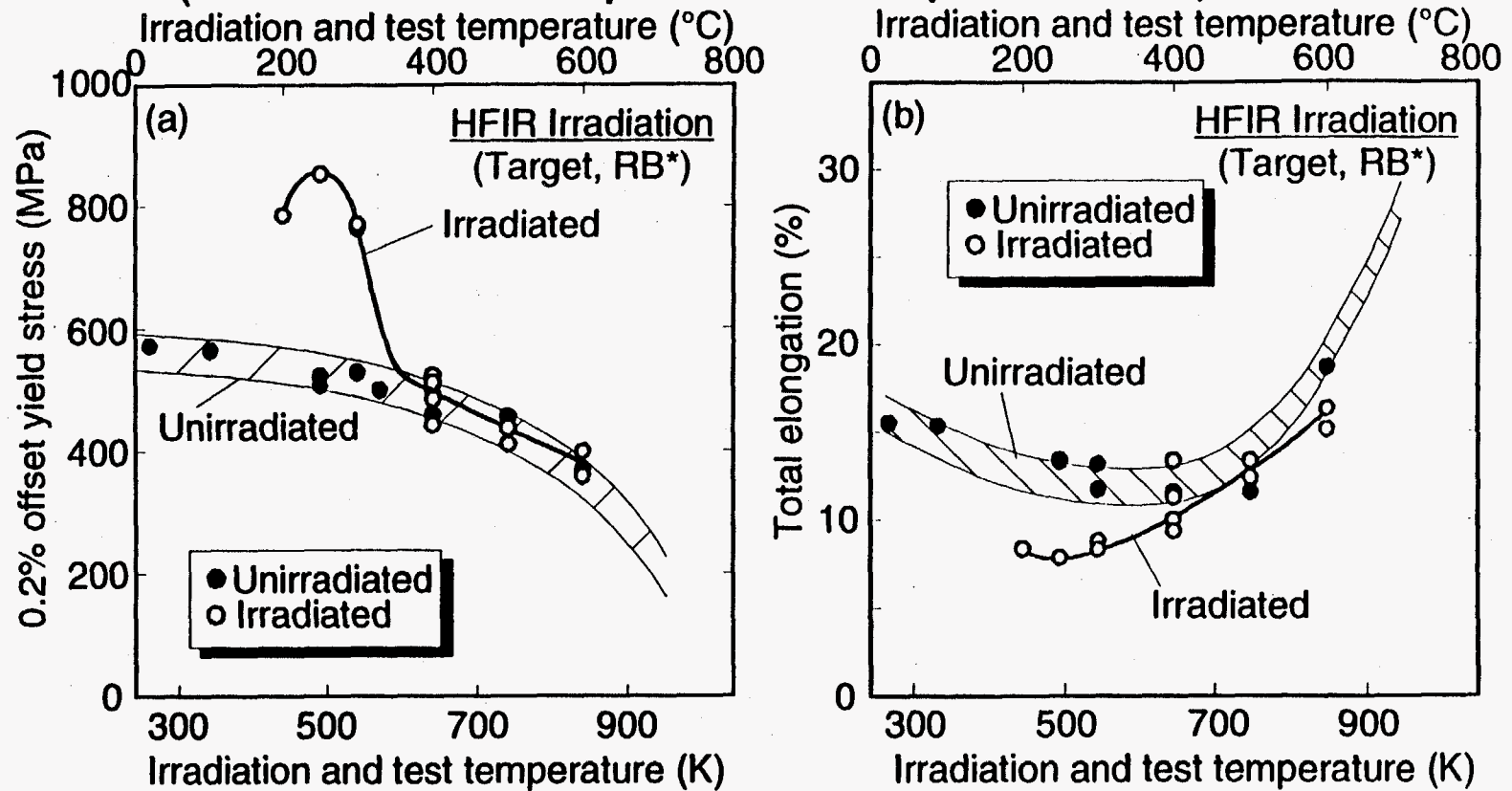
Absorbed Energy at 0°C after Aging (1st 5ton heat)



TTP Diagram

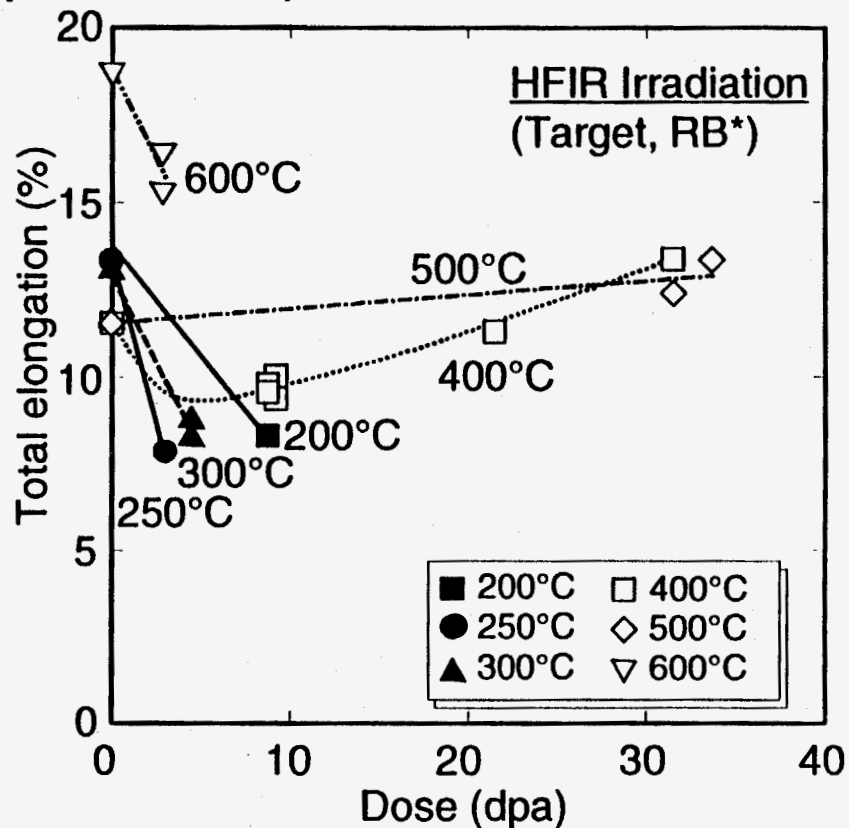
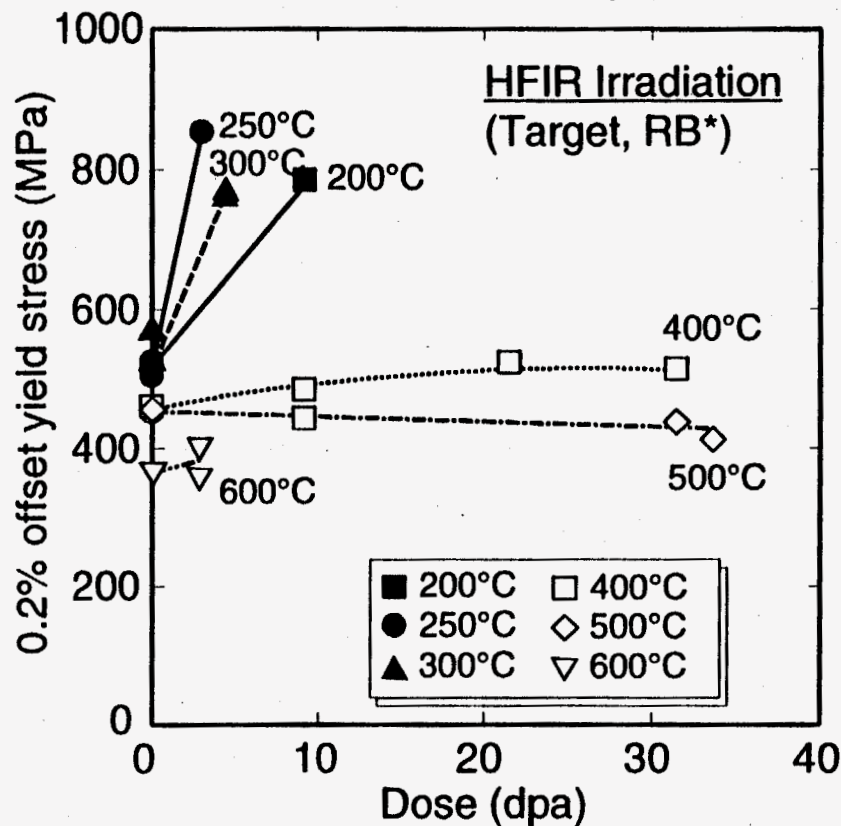


# TENSILE PROPERTIES AFTER HFIR IRRADIATION (Irradiation Temperature Dependence)



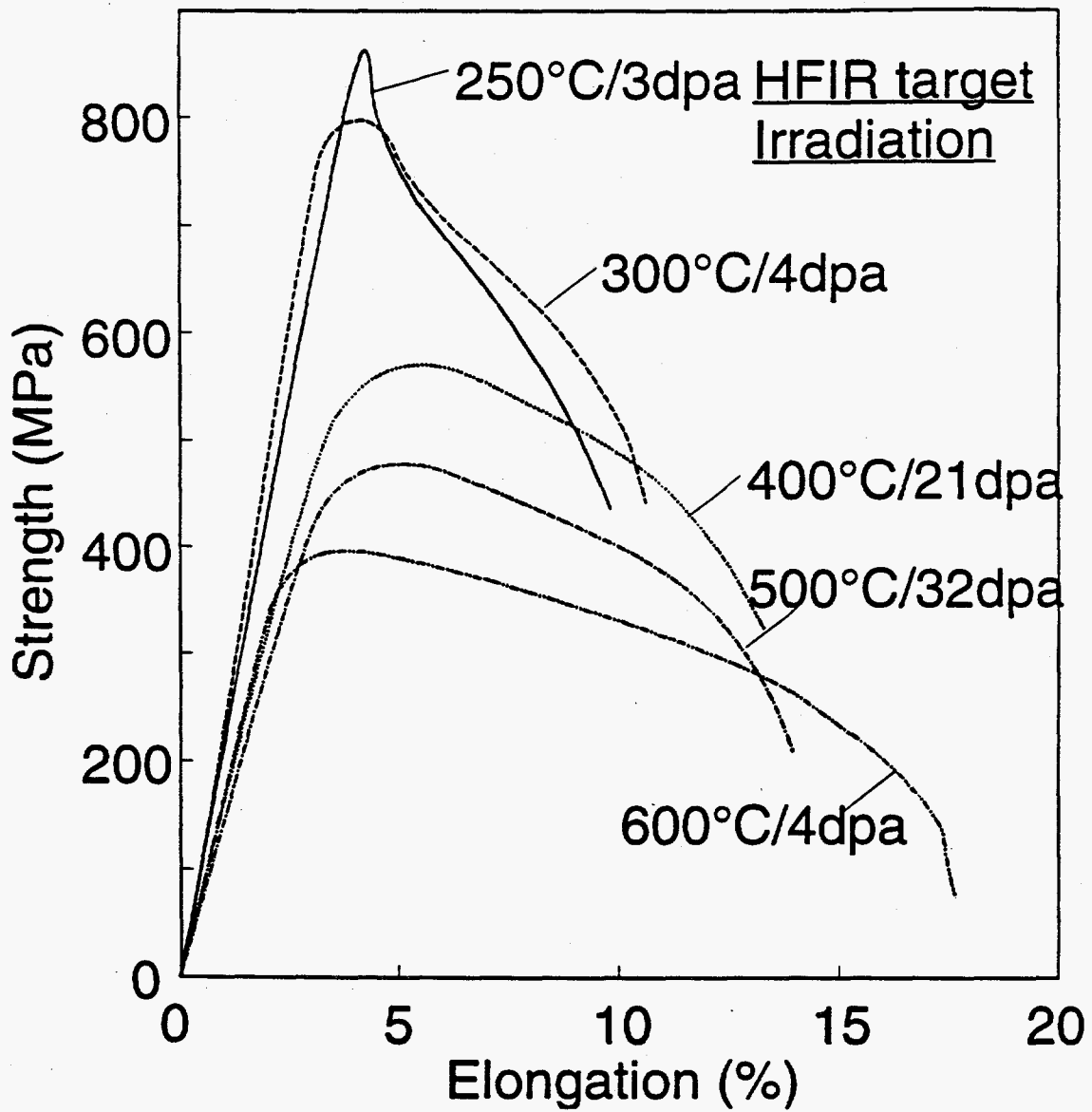
- Irradiation hardening has a peak at 250°C
- Almost no changes above 400°C

# TENSILE PROPERTIES AFTER HFIR IRRADIATION (Dose Dependence)



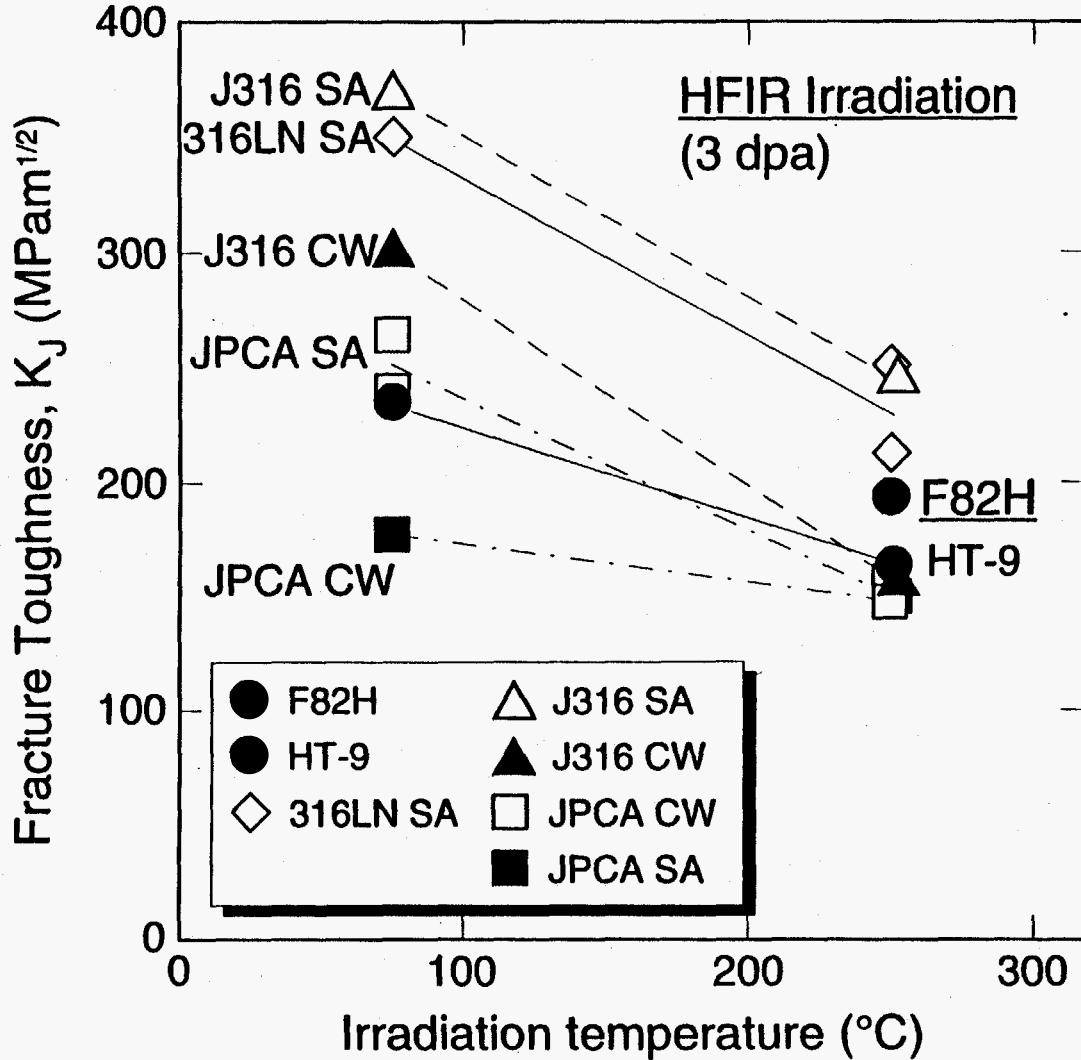
- Large Increase in YS between 200-300°C
- Elongation Increased after 10 dpa at 400°C

# TYPICAL STRESS-STRAIN CURVES AFTER HFIR IRRADIATION



# FRACTURE TOUGHNESS AFTER HFIR IRRADIATION

(0.18DCT specimen)



- 250C Irradiation: Irradiation Hardening DBTT Shift

- F82H has the similar level of Fracture Toughness to 316SS.

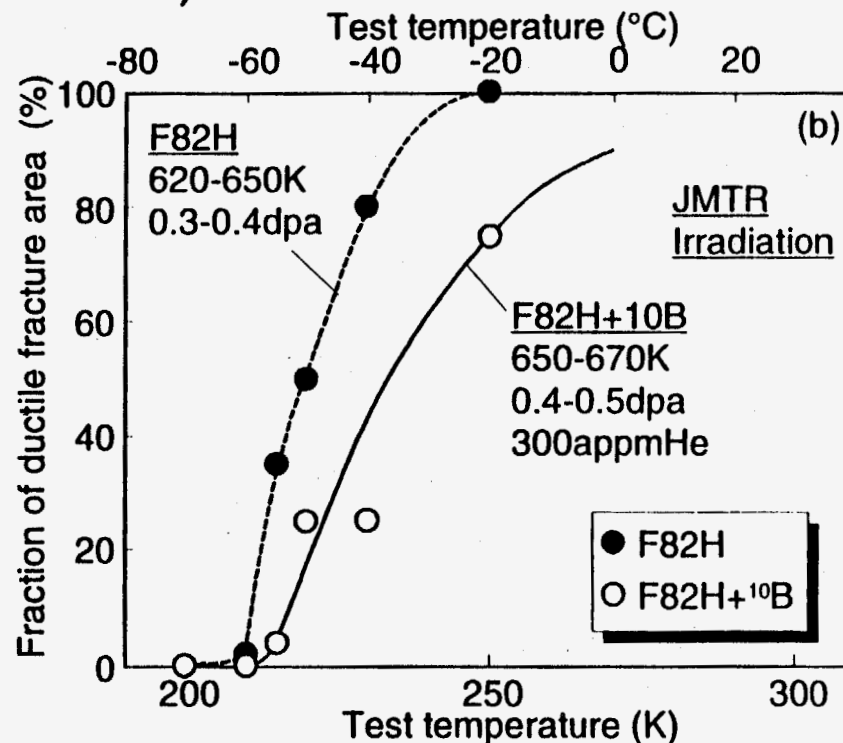
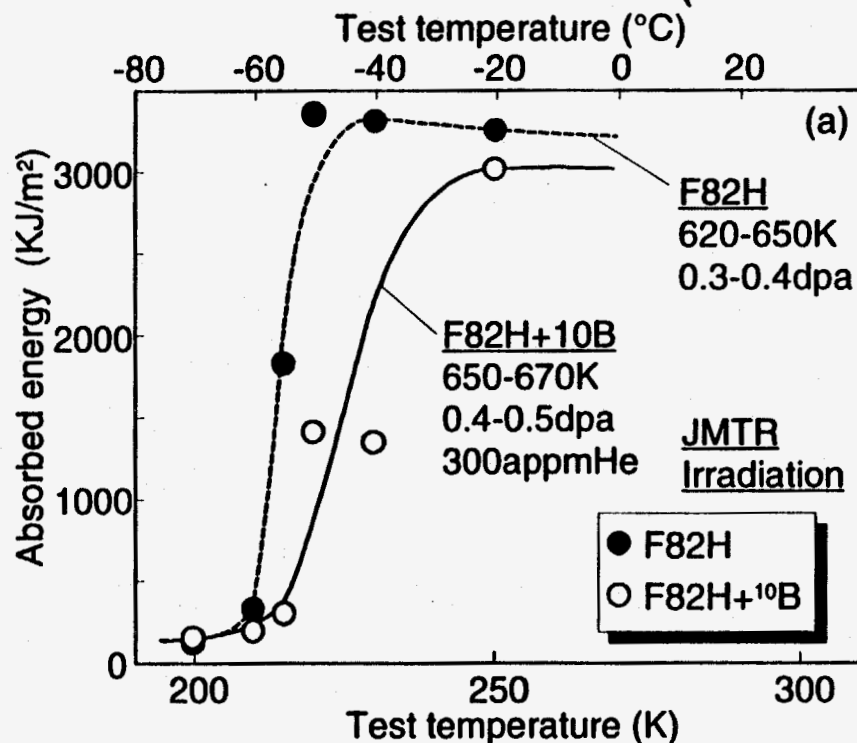
(250C, Unirrad.)

J316: 389 MPam<sup>1/2</sup>

F82H: 252 MPam<sup>1/2</sup>

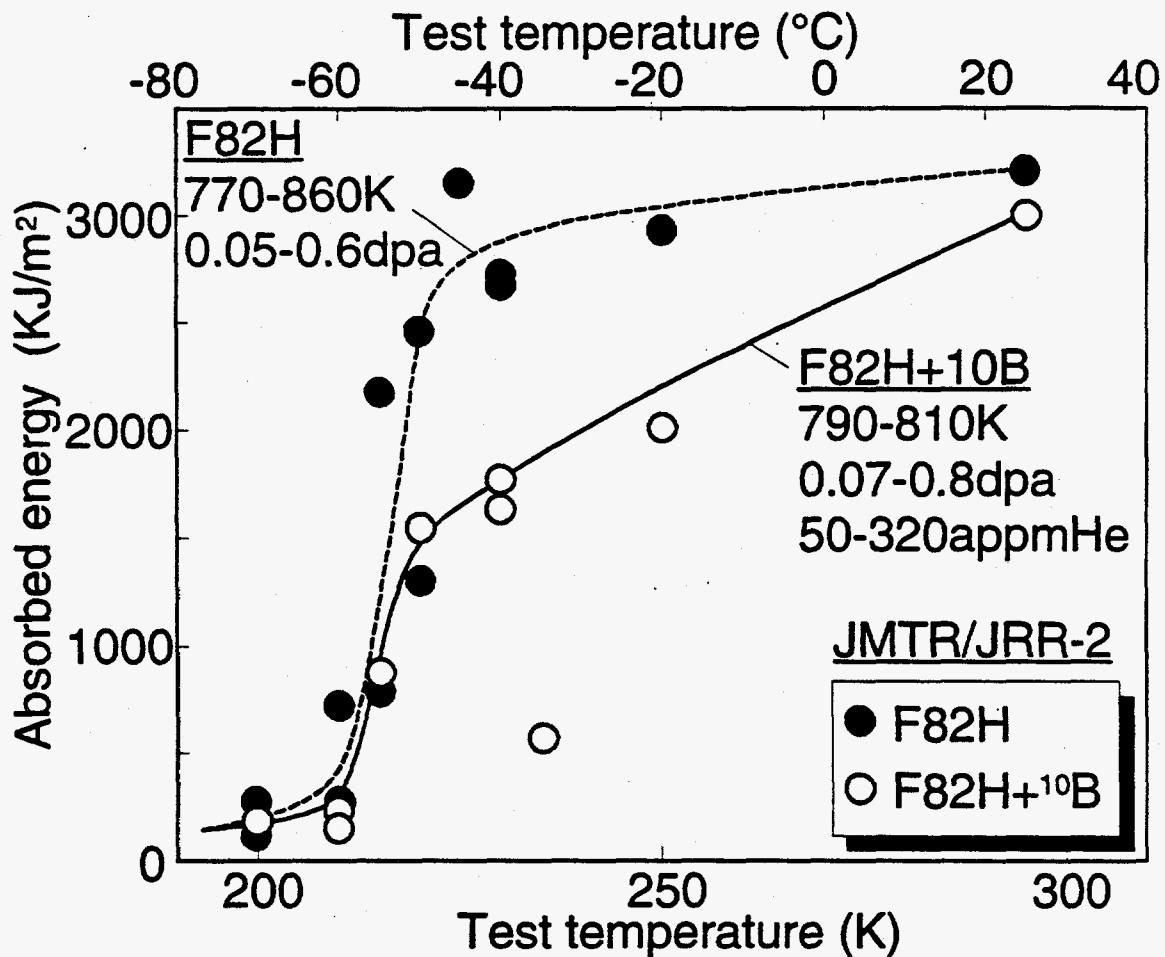
HT9 : 281 MPam<sup>1/2</sup>

# EFFECT OF HELIUM ON CHARPY IMPACT PROPERTY (JMTR: 400°C)

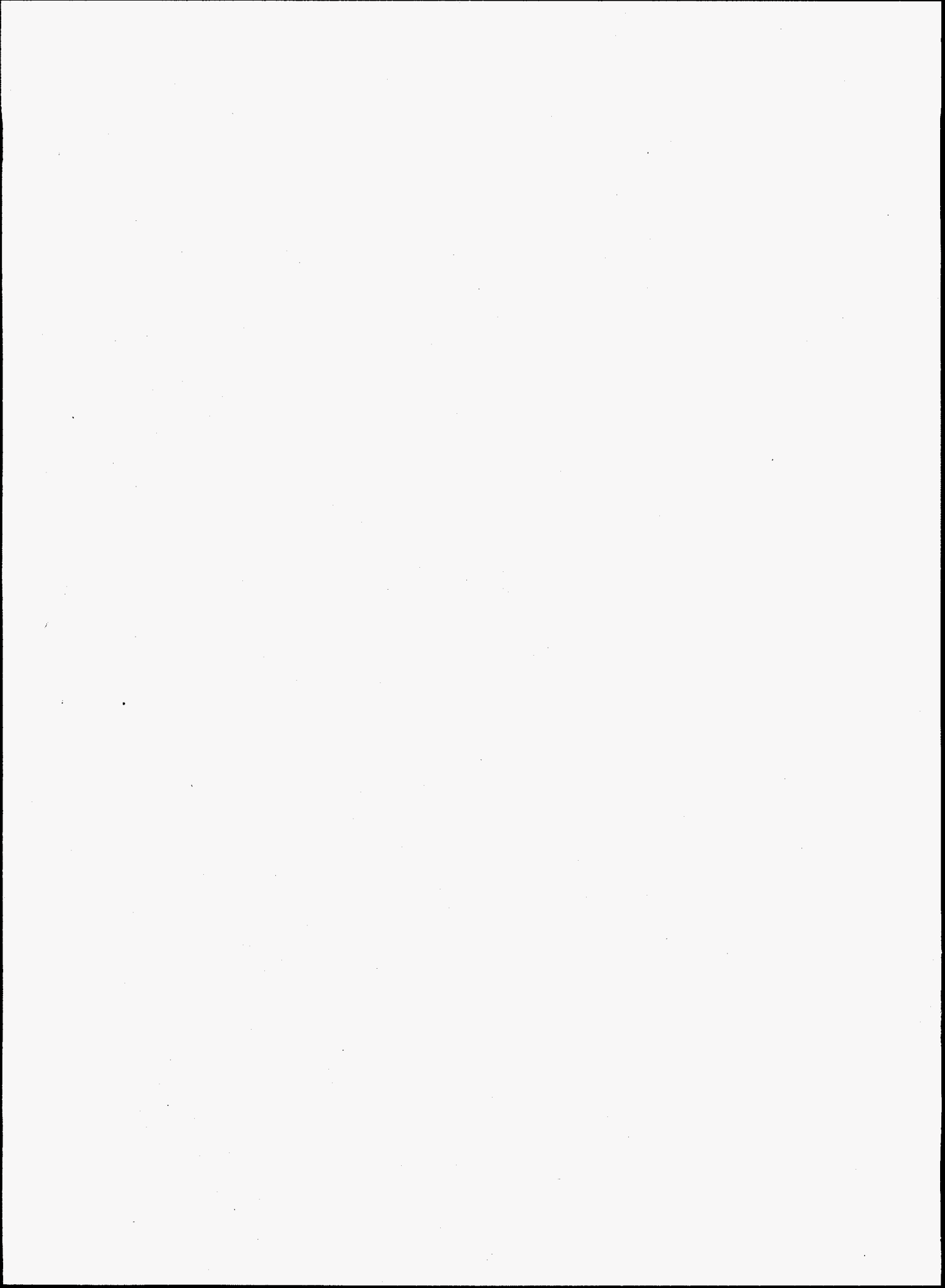


- <sup>10</sup>B-doped showed more increase in DBTT than B-free after 400°C irradiation. (~12°C)
- B-free had no DBTT shift by irradiation.

# EFFECT OF HELIUM ON CHARPY IMPACT PROPERTY (JMTR/JRR-2: 550°C Irradiation)



- <sup>10</sup>B-doped showed the difference in transition behavior after 550°C irradiation.
- The same transition behavior was observed on the 50 and 320appmHe specimens.



**IEA Workshop on Ferritic Steels  
Culham, UK, October 24-25, 1996**

**MIXED MODE FRACTURE TOUGHNESS  
IN F82H**

**Huaxin Li  
and  
D. S. Gelles**



**Pacific Northwest National Laboratory**



# F82H MIXED MODE FRACTURE TOUGHNESS

## ■PURPOSE

TO DETERMINE THE CONSEQUENCE OF CRACK ANGLE ON FRACTURE TOUGHNESS, BETWEEN MODE I AND III

## ■APPLICABILITY

THE RESULTS PREVIOUSLY OBTAINED ARE FOR A SMALL HEAT (#8033) FROM NKK WITH A DIFFERENT HEAT TREATMENT FROM THE JAERI RECOMMENDATION. COMPARISON WITH WITH JAERI HEAT IN THE RECOMMENDED TREATMENT (25 mm PLATE) ARE PRESENTED.

## ■COMPOSITION

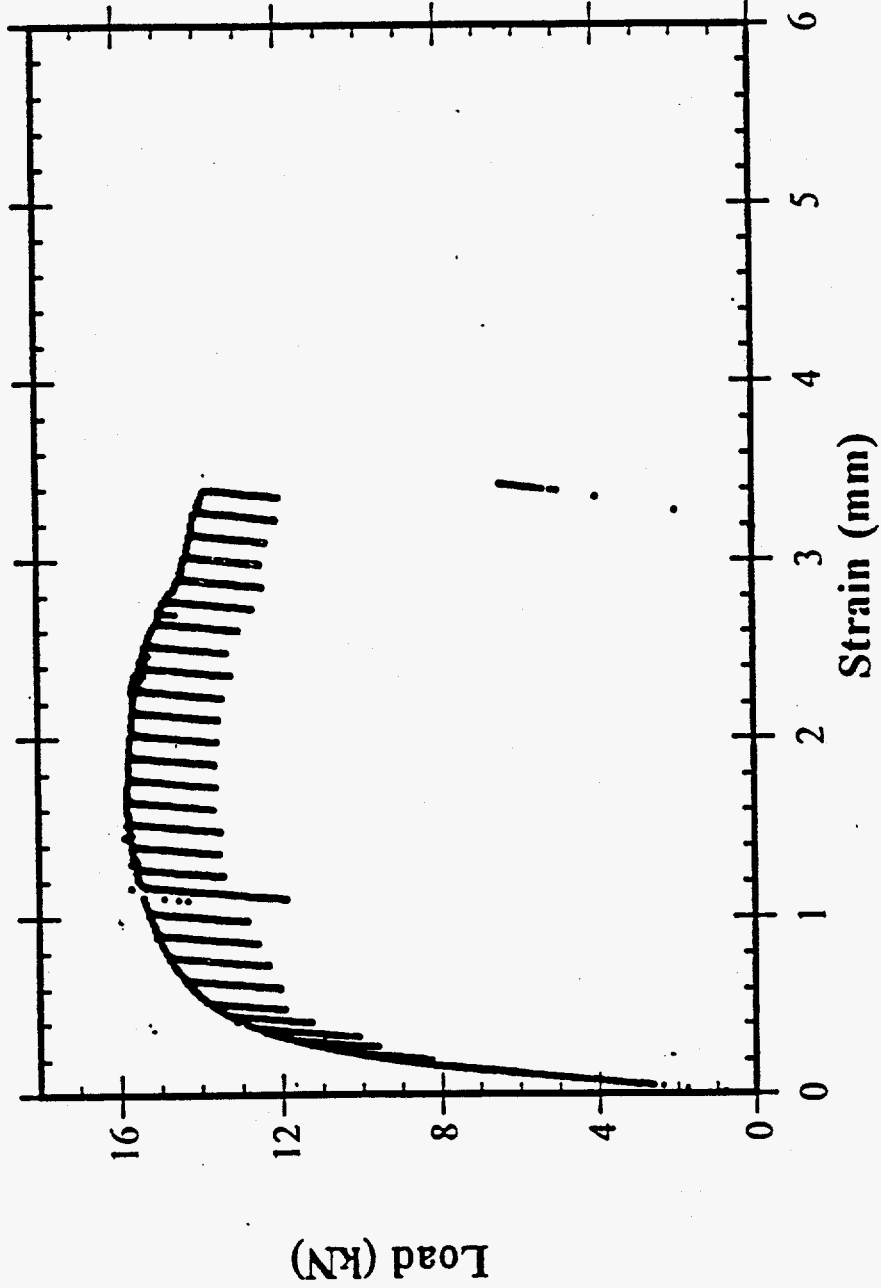
C	Cr	W	V	Ta	Mo	P	S	Si	Mn
0.096	7.71	2.1	0.18	0.04	<0.01	0.003	0.003	0.10	0.15
Cu	Ti	Ni	Nb	SolAl	B	T.O	T.N	Fe	
0.01	<0.1	0.008	<0.005	0.005	0.0004	0.0043	0.0028	balance	

## ■HEAT TREATMENT

1000°C/20h/AC + 1100°C/7min/ac + 700°C/2h/AC

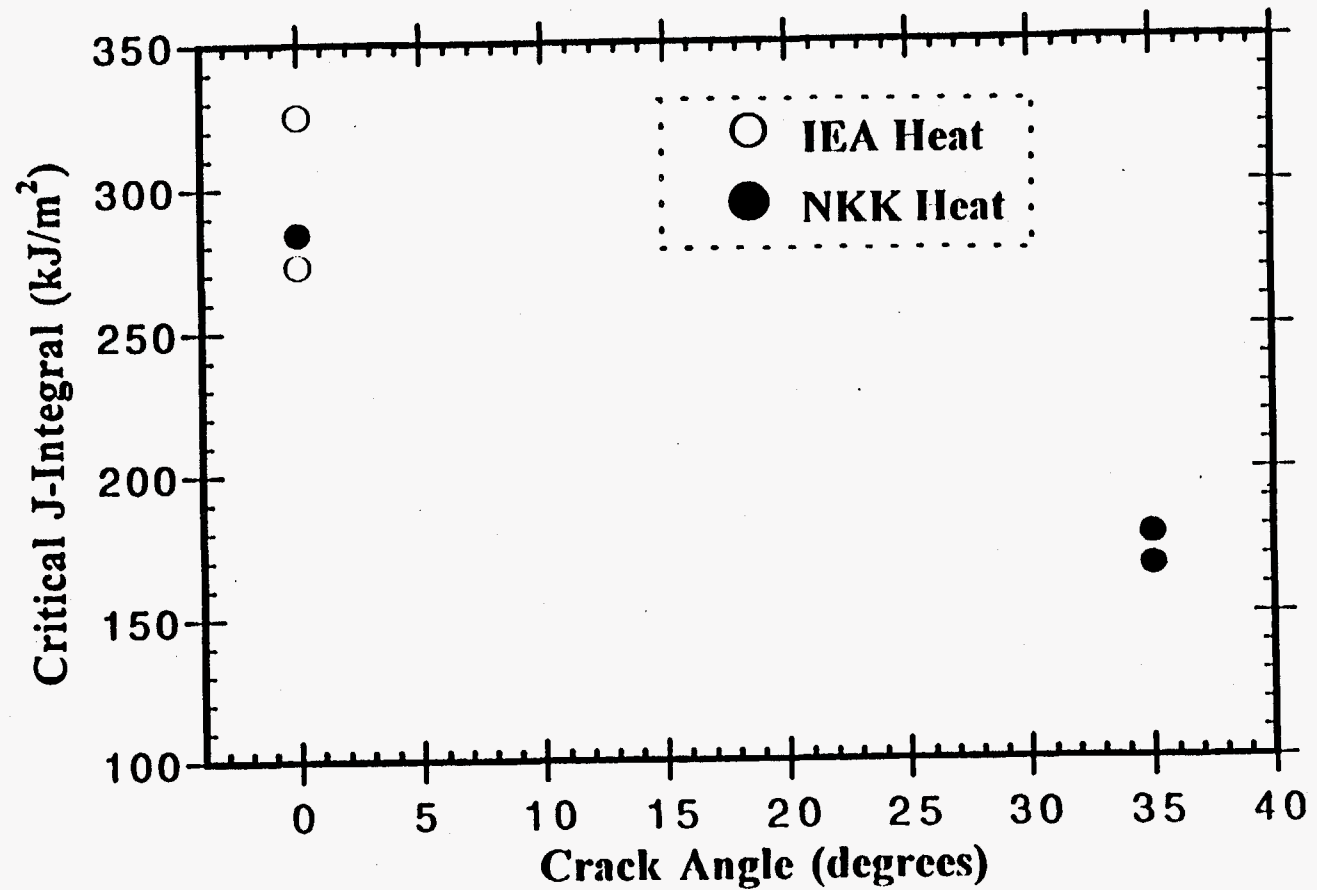
# IEA F82H MIXED MODE RESPONSE

Mixed-Mode I/III Specimen (35 degree, IEA-9)

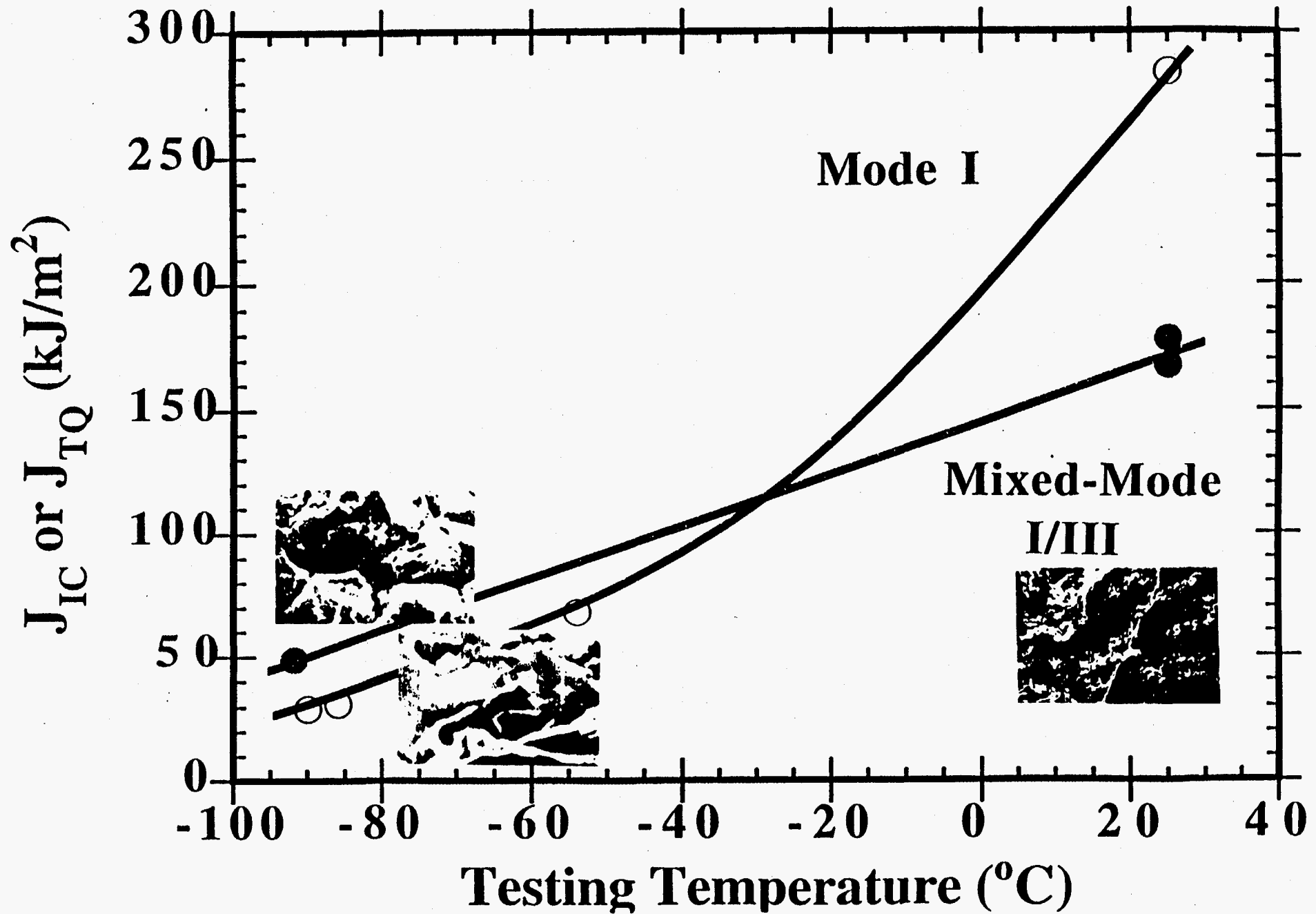


## RECENT RESULTS

- NO HEAT TO HEAT / HEAT TREATMENT DIFFERENCES FOR MODE I FRACTURE TOUGHNESS

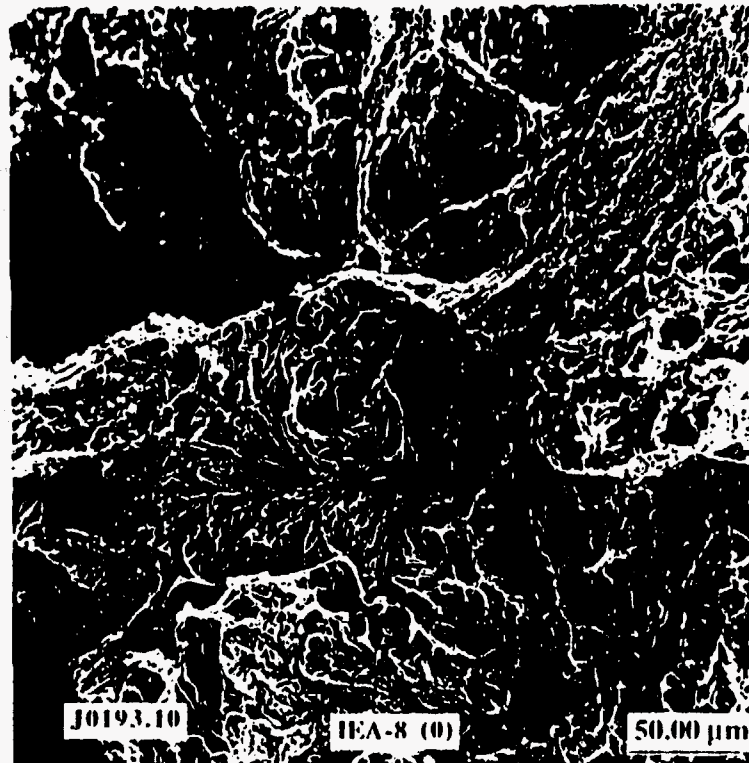


- TWO MIXED MODE TESTS GAVE UNEXPECTED RESULTS



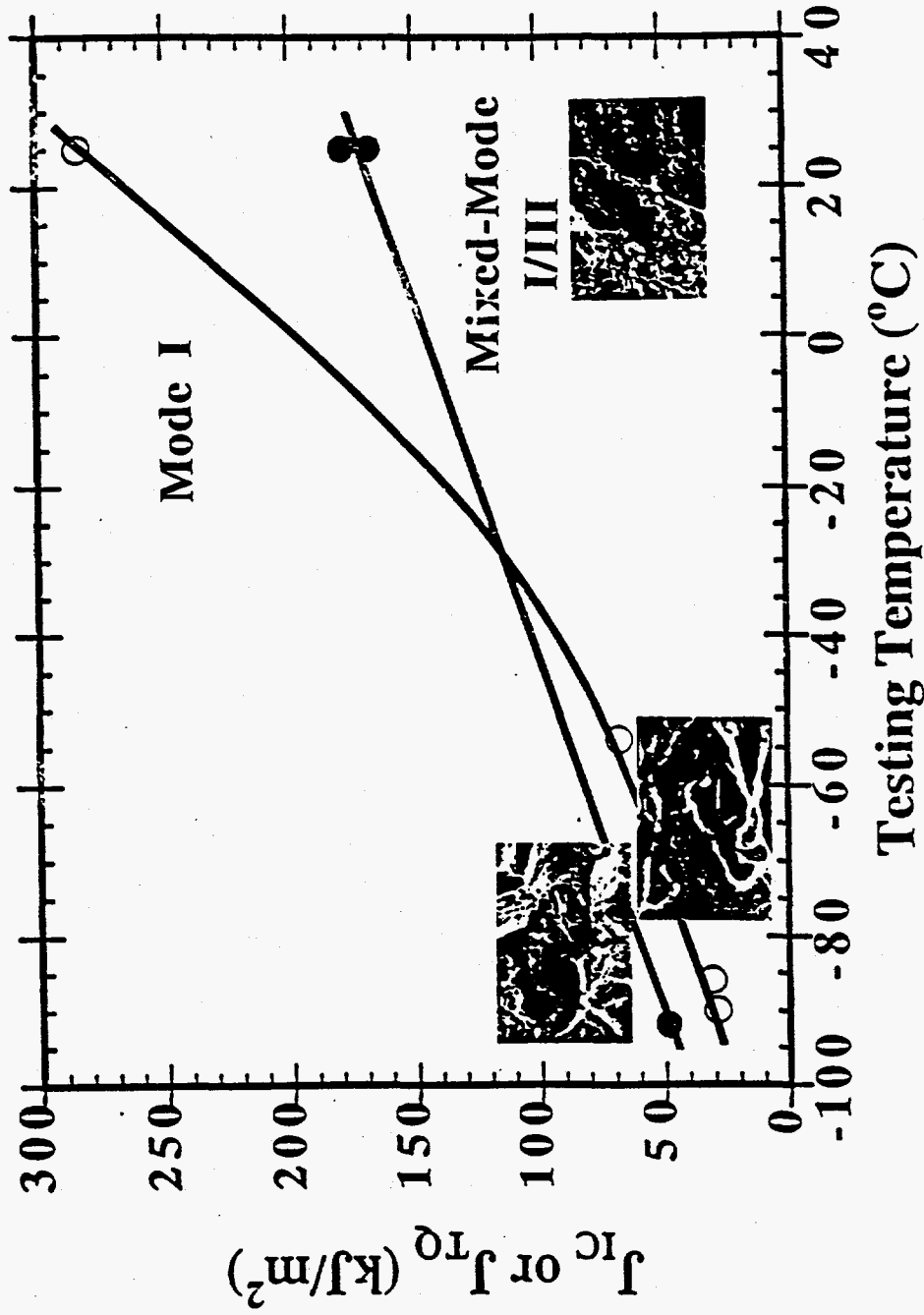
## IEA F82H MIXED MODE RESPONSE

- FRACTURE SURFACES SHOW DIMPLE RUPTURE FOLLOWED BY CLEAVAGE FRACTURE ON A PLANE APPROXIMATELY 15° TOWARDS MODE I
- EXAMPLES OF LARGE INCLUSIONS WERE FOUND



# IEA F82H MIXED MODE RESPONSE IS UNEXPECTED

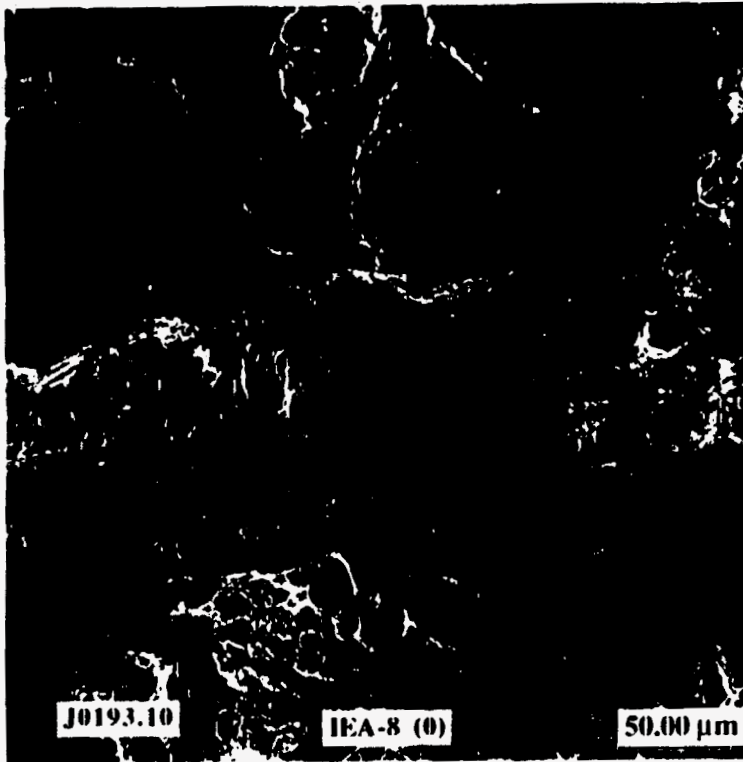
■ MIXED MODE GIVES INTERGRANULAR FRACTURE ON LOWER SHELF



■ BEHAVIOR OBSERVED SUGGESTS A HIGH DBTT

## IEA F82H MIXED MODE RESPONSE

- FRACTURE SURFACES SHOW DIMPLE RUPTURE FOLLOWED BY CLEAVAGE FRACTURE ON A PLANE APPROXIMATELY  $15^\circ$  TOWARDS MODE I
- EXAMPLES OF LARGE INCLUSIONS WERE FOUND



## **CONCLUSIONS**

- **MODE I FRACTURE TOUGHNESS TESTS ON HEAT TO HEAT / HEAT TREATMENT VARIABLE INDICATES NEGLIGIBLE EFFECTS**
- **MIXED MODE I/III TESTS ON THE IEA F82H 25 mm PLATE GAVE UNEXPECTED RESULTS. LARGE INCLUSIONS ARE INDICATED. TESTS WILL BE CONTINUED TO PROVIDE EXPLANATION.**



**Miscellaneous Results on F82H: Effects of Ti, N, Ta  
on Grain Size, Vacuum Properties, etc.**

**A. Hishinuma and K. Shiba  
Material Science & Engineering Dep.  
JAERI, Tokai**

**IEA Workshop on LAM  
Culham, U.K.  
October 24, 25, 1996**

- 1. Effects of Ta, Ti, N on DBTT of F82H**
- 2. Radiochemical Analysis of Impurities in F82H**
- 3. Vacuum properties-Gas Desorption properties of F82H**
- 4. Corrosion Behavior of F82H**

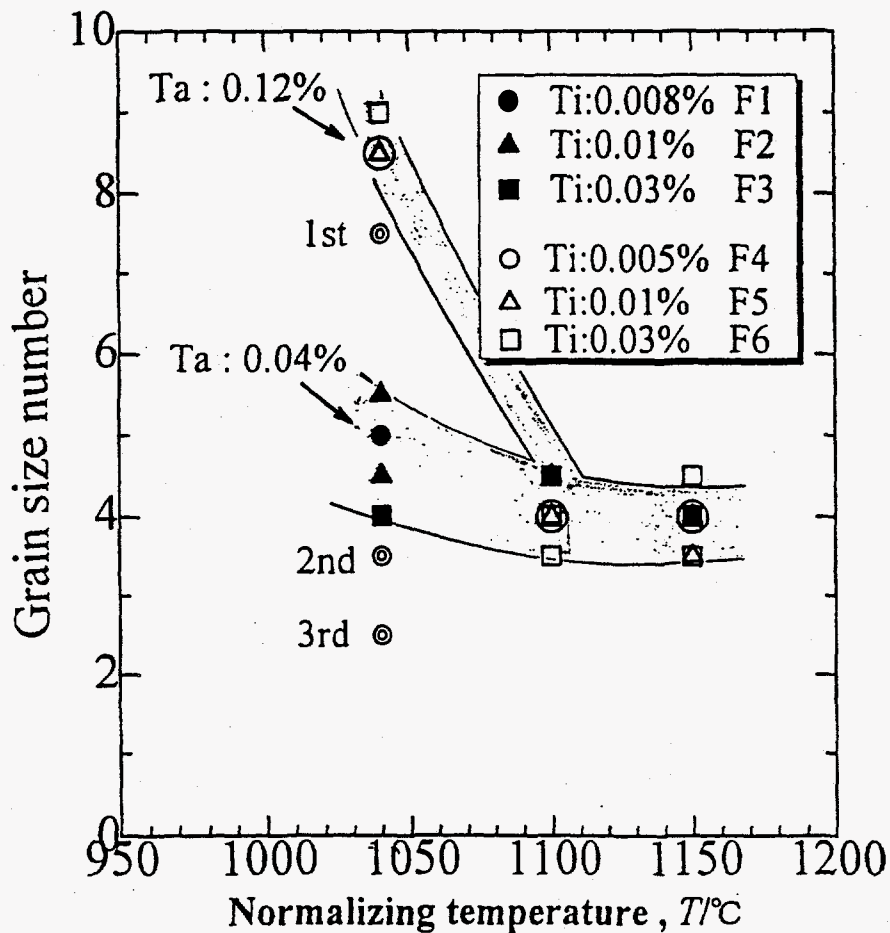
# 1. Effects of Ta, Ti and N on DBTT of F82H

## (1) Samples used

合金	C	Si	Mn	P	S	Cr	W	V
F1	0.095	0.11	0.49	0.0012	0.0032	7.83	1.91	0.19
F2	0.095	0.11	0.49	0.0010	0.0032	7.86	1.92	0.19
F3	0.096	0.11	0.48	0.0011	0.0033	7.88	1.91	0.19
F4	0.094	0.11	0.49	0.0010	0.0033	8.08	1.96	0.20
F5	0.095	0.12	0.50	0.0008	0.0032	8.02	1.95	0.20
F6	0.096	0.12	0.49	0.0007	0.0033	8.01	1.96	0.20

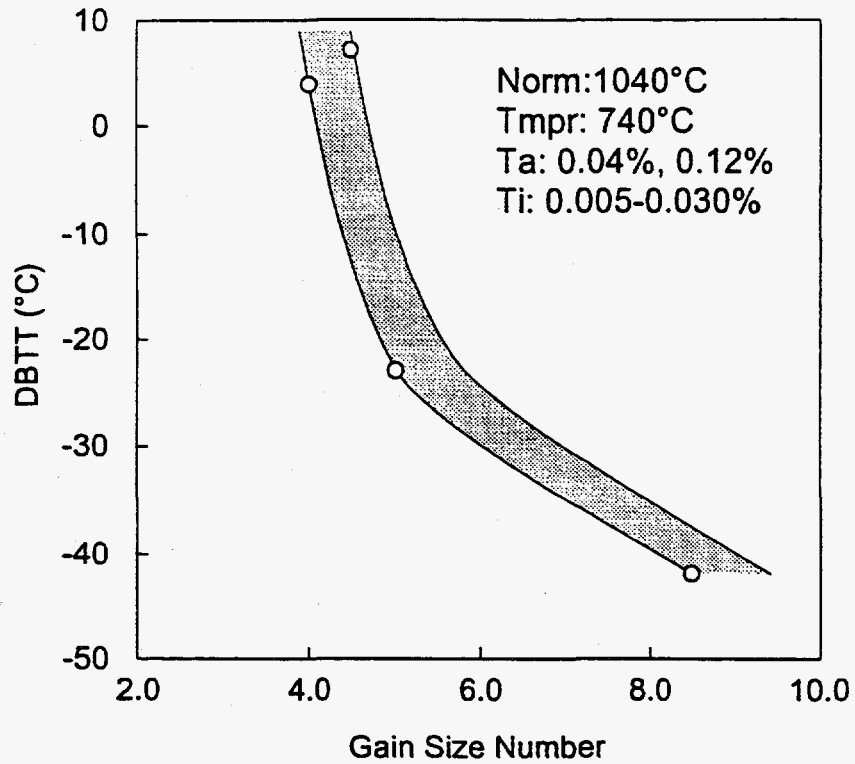
合金	Ta	Ti	T.N.	Sol.Al	Total.Al	O	Fe
F1	0.04	0.008	0.0082	0.012	0.017	0.0035	bal.
F2	0.04	0.010	0.0084	0.014	0.020	0.0027	bal.
F3	0.04	0.030	0.0093	0.014	0.019	0.0029	bal.
F4	0.12	0.005	0.0092	0.014	0.020	0.0039	bal.
F5	0.12	0.010	0.0095	0.016	0.018	0.0038	bal.
F6	0.12	0.030	0.0097	0.015	0.019	0.0034	bal.

## (2) Effects of Normalizing temp. on Grain size



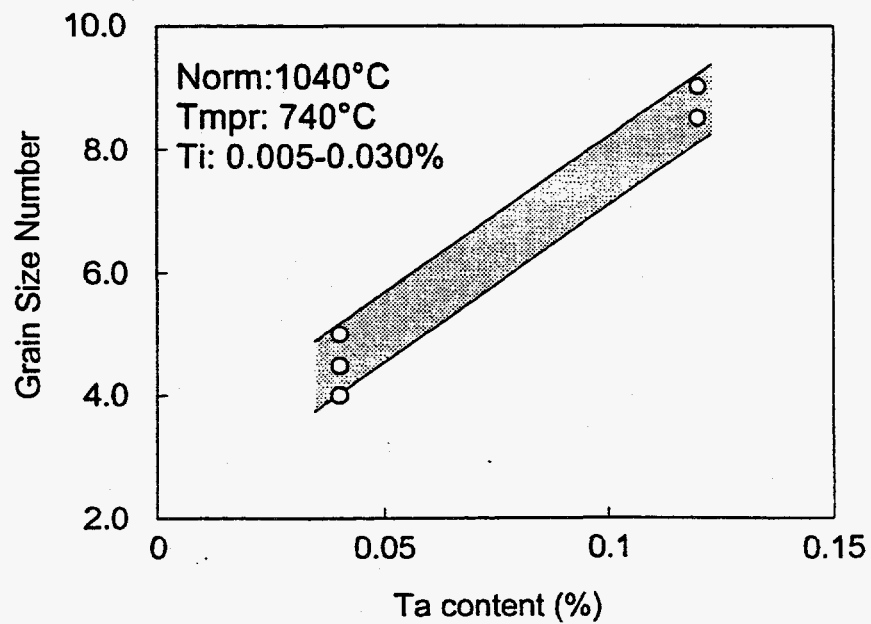
**(3) Effects of Grain size on DBTT**

- DBTT strongly depends on grain size;



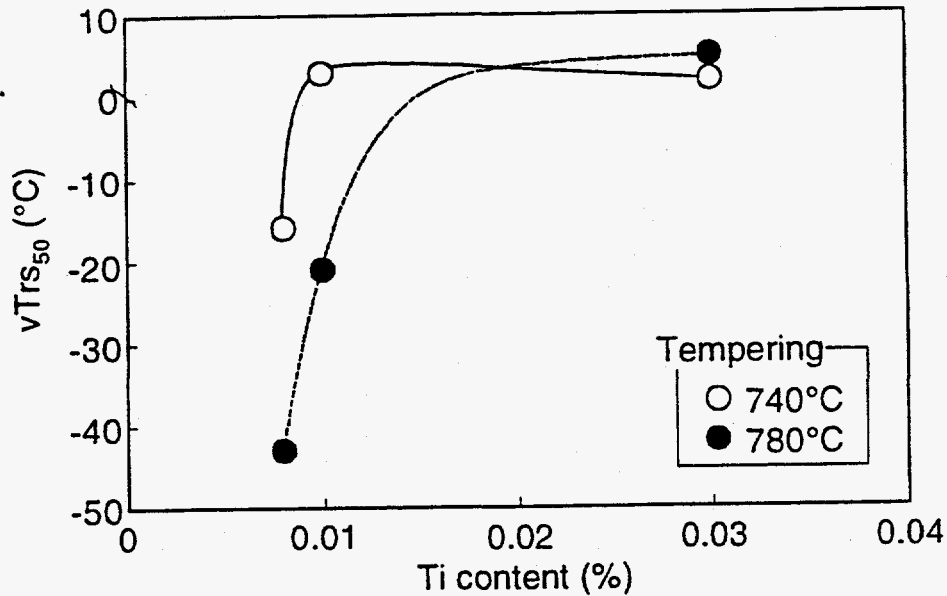
**(4) Effects of Ta on Grain size**

- Ta content gives large effects on grain size



### (5) Effects of Ti on DBTT

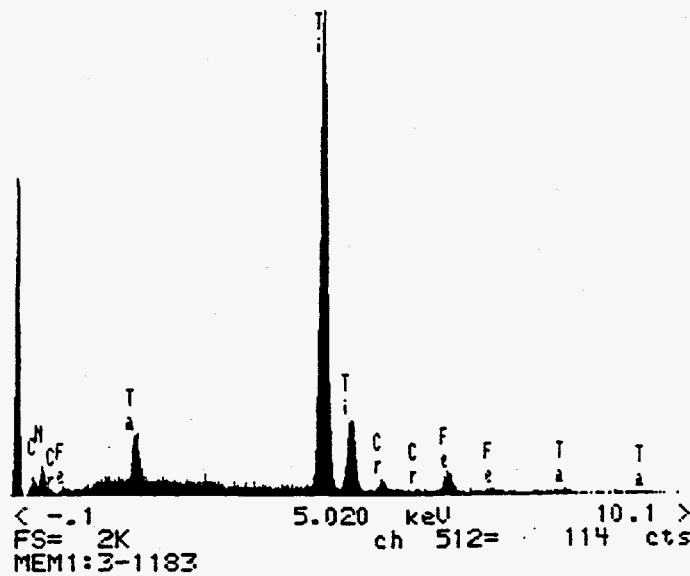
- Ti content gives large effect of DBTT behavior
- It is important to keep Ti content less than 0.01% or N content less than 0.004% to prevent Ti(N,C) precipitation



Relationship between DBTT and Ti content (0.04%Ta, 1040°C Normalizing)

### (6) Ta precipitates

- Ta was detected in Ti(N,C) precipitates or Ti related inclusions



SEM-EDX analysis of inclusions

## **(6) Conclusions**

- 1) DBTT of ferritic steels depends strongly on grain size ; smaller grain size shows lower DBTT.**
- 2) The grain size decreases with increase in Ta content, and therefore DBTT decreases with Ta content**
- 3) Ta co-precipitates with Ti(N,C), which precipitates when Ti content is larger than 0.01% or N content larger than 0.04%.**
- 4) The difference in DBTT among large heats is considered to be effects of nitrogen which may easily form TiN(Ta) and decrease in Ta solution in the matrix; the 1st heat contain small amount of N compared with 2nd and 3rd heats and then, their DBTT show low. .**

## 2. Radiochemical Analysis of Impurities in F82H

### Impurity analysis of F82H after fusion neutron irradiation in FNS (Preliminary results)

#### (1) Neutron Irradiation Experiments

Irradiation time : 7.25 days

Total fluence :  $2.70 \times 10^{16} / \text{cm}^2$  ( $E_n=14.9$  MeV)

Cooling time : 214 days

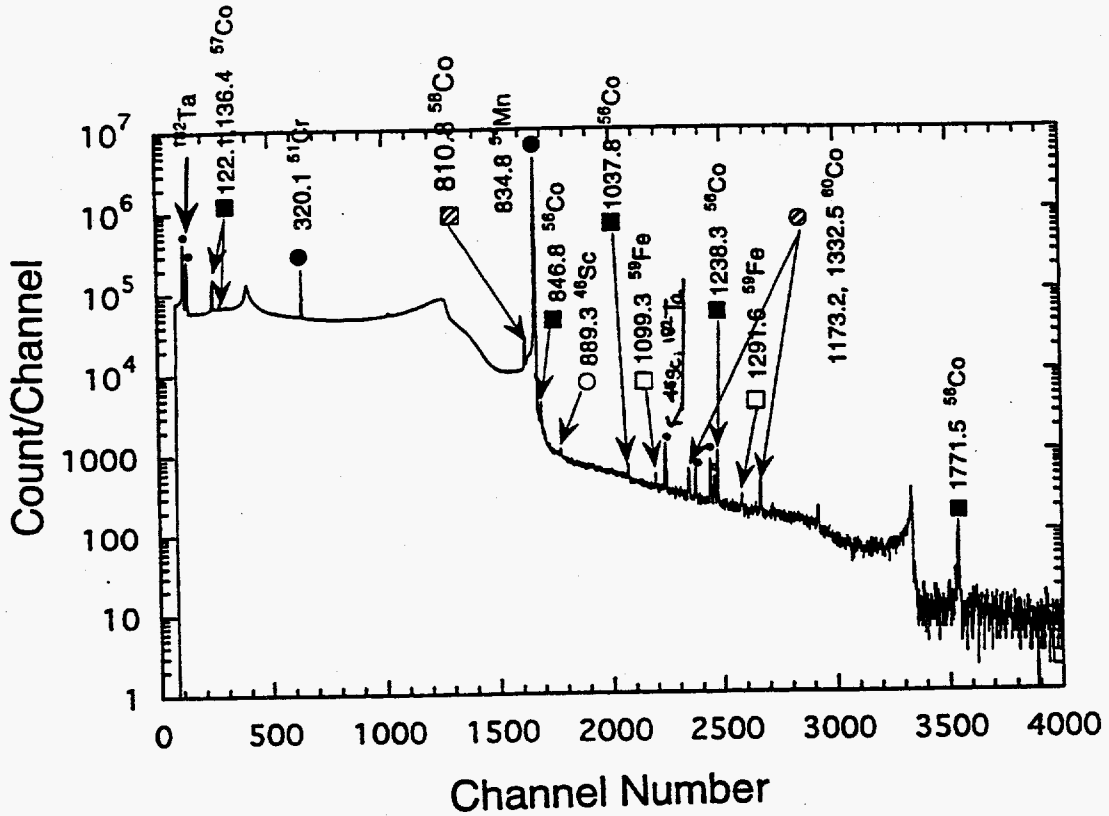
Measuring time : 1428 hr

#### (2) Nuclear reactions and nuclear data

- Radioactivity from  $^{54}\text{Mn}$  is the maximum after cooling time of about a half-year, which is generated mainly from  $^{54}\text{Fe}$ . The result corresponds the calculated one.
- The relatively large radioactivity came from  $^{51}\text{Cr}$  and  $^{57}\text{Co}$ .
- Other nuclides with high radioactivity from impurities were not detected at this stage.

Radio-nuclide	half life (y)	$\gamma$ ray (KeV)	Emmission Rate (%)	Radioactivity (Bq)	Activation Reaction	Cross Section (mb)
46Sc	83.83 d	889.3	99.984(1)	$0.015 \pm 0.003$	46Ti(n,p) 47Ti(n,np)	210(14) 80.0(59)
51Cr	27.704 d	320.1	99.83(14)	$6.2 \pm 0.2$	50Cr(n, $\gamma$ ) 52Cr(n,2n) 54Fe(n, $\alpha$ )	376(30) 89.1(62)
54Mn	312.20 d	834.8	99.975(2)	$240 \pm 7$	55Mn(n,2n) 54Fe(n,p)	803(45) 272(13)
59Fe	44.496 d	1099.3	56.5(15)	$0.009 \pm 0.002$	58Fe(n, $\gamma$ ) 59Co(n,p)	457(41)
56Co	77.7 d	846.8	99.9	$0.098 \pm 0.005$	56Fe(p,n) <sup>2)</sup>	
57Co	271.77 d	122.1	85.5(4)	$3.2 \pm 0.1$	58Ni(n,np)	66.1(35)
58Co	70.916 d	810.8	99.5(3)	$0.56 \pm 0.02$	59Co(n,2n) 58Ni(n,p)	801(42) 269(15)
60Co	5.271 y	1173.2	99.90(2)	$0.018 \pm 0.002$	59Co(n, $\gamma$ ) 60Ni(n,p)	103.3(69)
182Ta	115.0 d	1121.4	27.3(5)	$0.036 \pm 0.002$	181Ta(n, $\gamma$ ) 182W(n,p) 183W(n,np)	6.65 <sup>3)</sup> <0.34 <sup>4)</sup>

#### (4) Measured $\gamma$ -Ray Spectrum

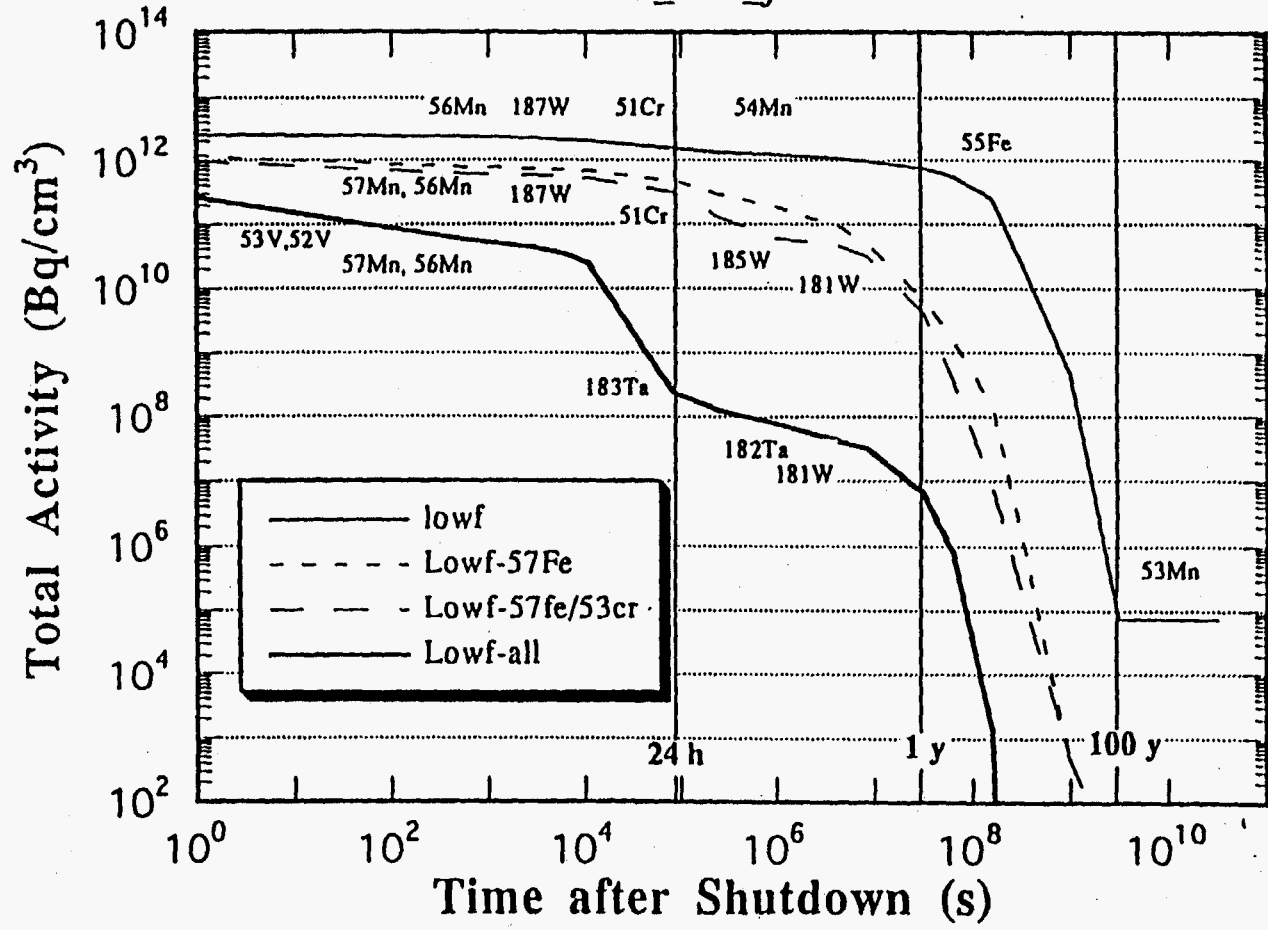


#### (5) Results of Radiochemical Analysis

- Impurities in F82H are within a range or less than limitational values

Elements	Radiochemical Analysis (wt.%)	Range of F82H (wt.%)
Ti	$0.0057 \pm 0.0011$	0.004 - 0.012
Mn	$0.10 \pm 0.02$	0.05 - 0.20
Ni	$0.021 \pm 0.001$	< 0.10
Co	$0.0020 \pm 0.0005$	< 0.01

low\_act\_f





### 3. Gas Desorption Properties of F82H (by K. Suzuki. et al, (Hokkaido U))

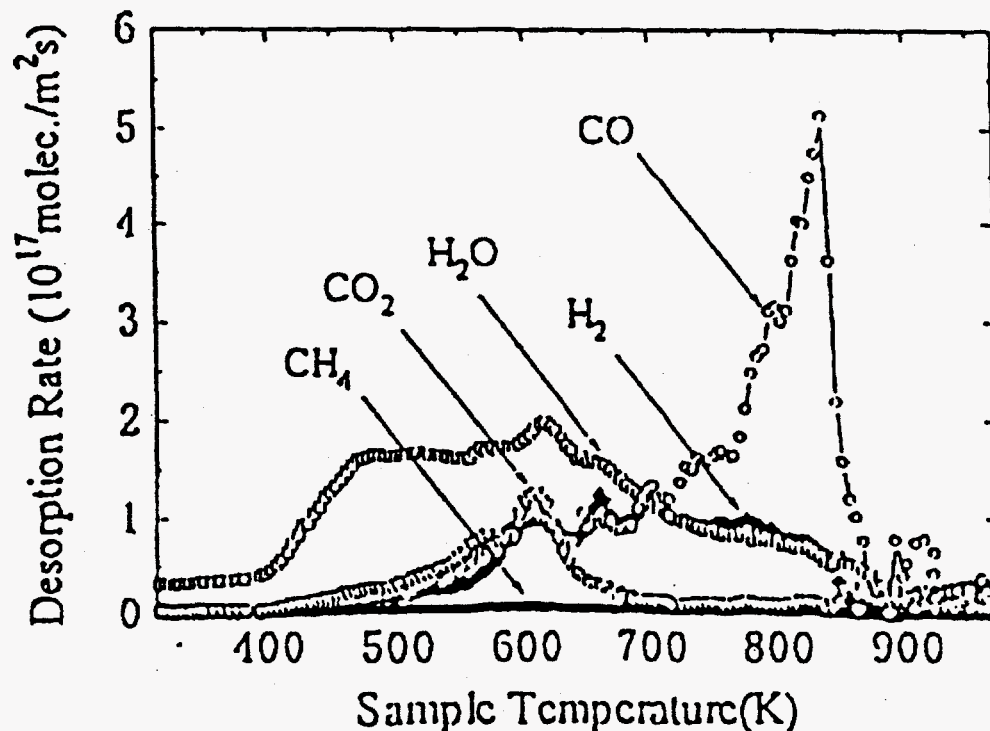
To investigate gas desorption properties and change in surface compositions

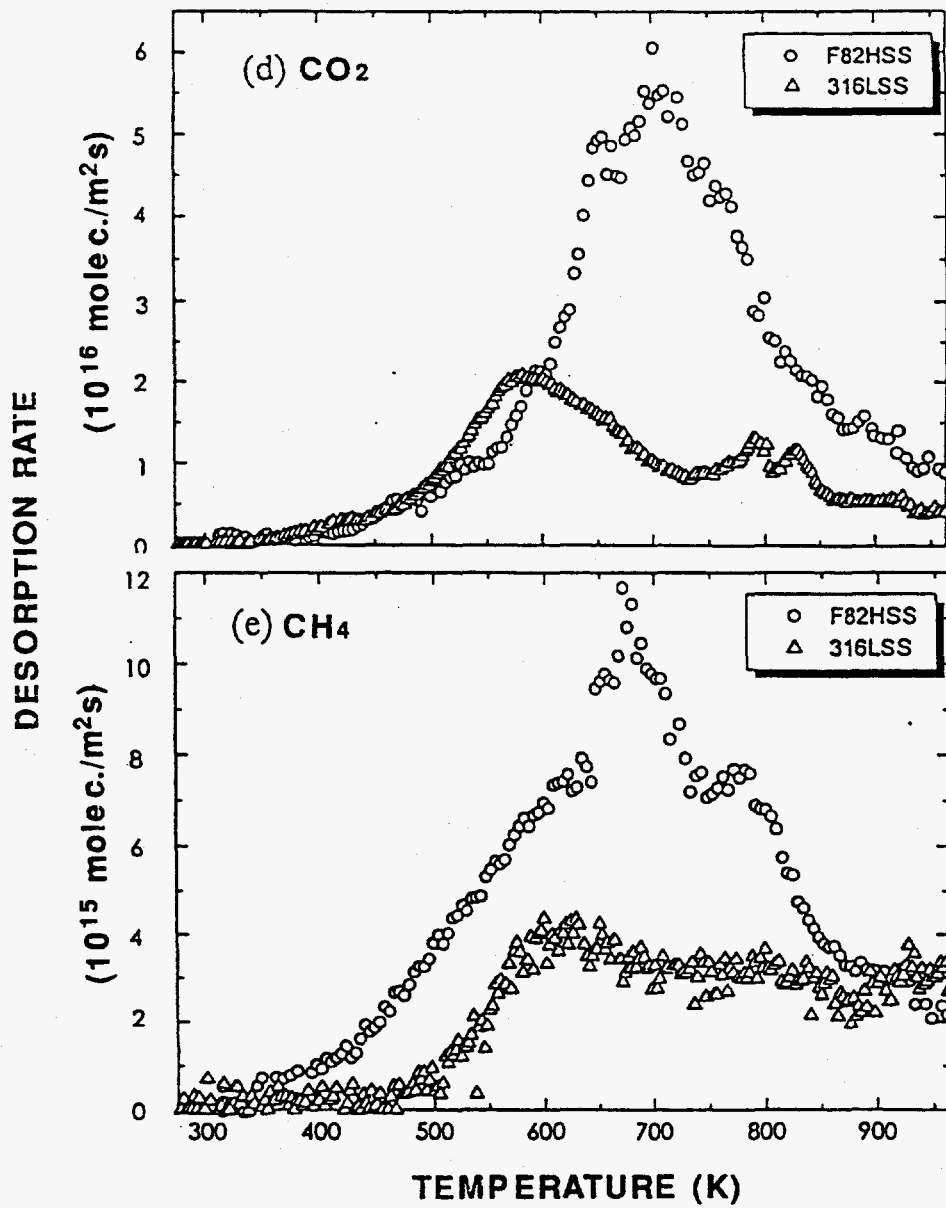
#### (1) TDS spectra for major gas species

- Major gas species desorped are CO, H<sub>2</sub>O, CO<sub>2</sub>, H<sub>2</sub>, C<sub>x</sub>H<sub>x</sub> in order of desorption volume.
- The desorption of H<sub>2</sub>O was firstly observed at 400 K, and that of all species were observed at 600 K. Around 850 K, a large amount of CO desorption was observed.
- The sum of CO and H<sub>2</sub>O is larger than 80 % of total desorption volume
- Two or three peaks in TDS spectra were observed in all of the gas species.

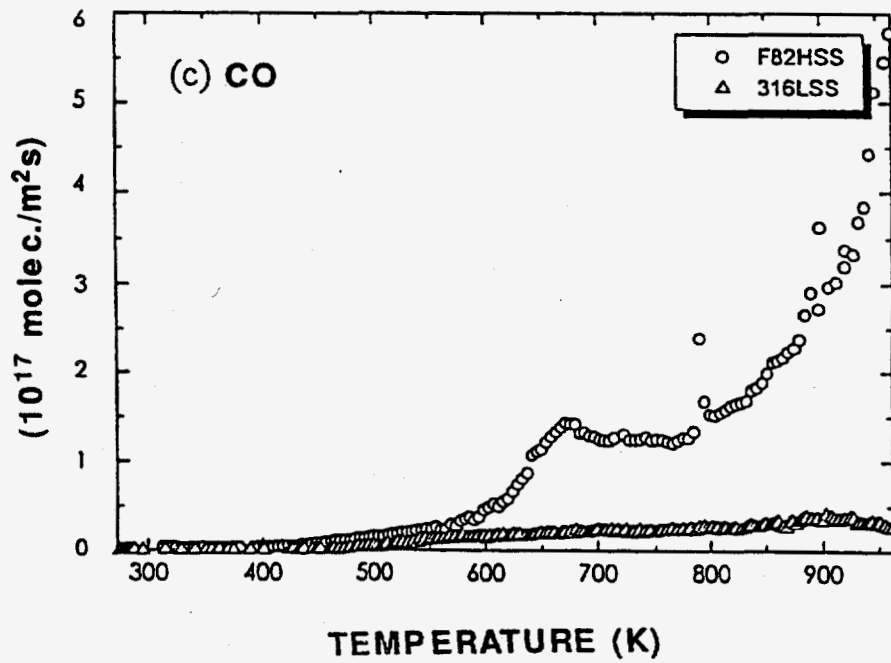
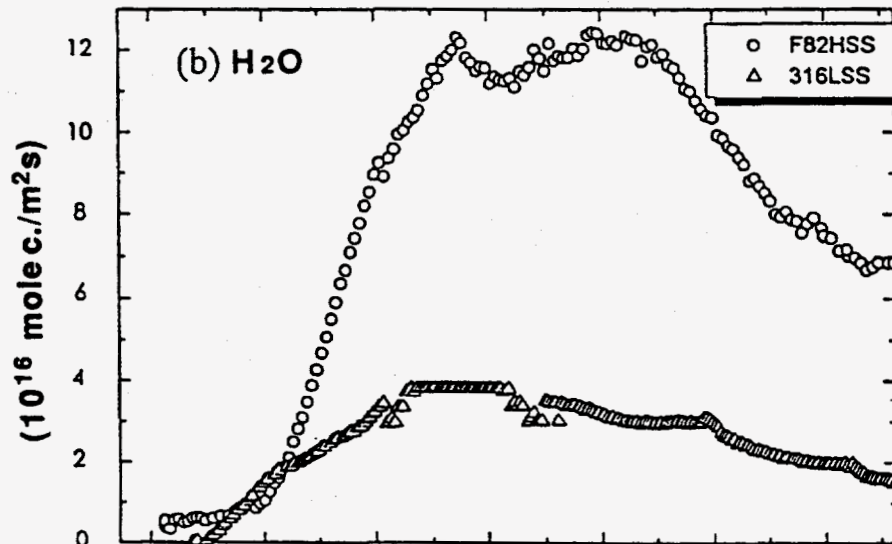
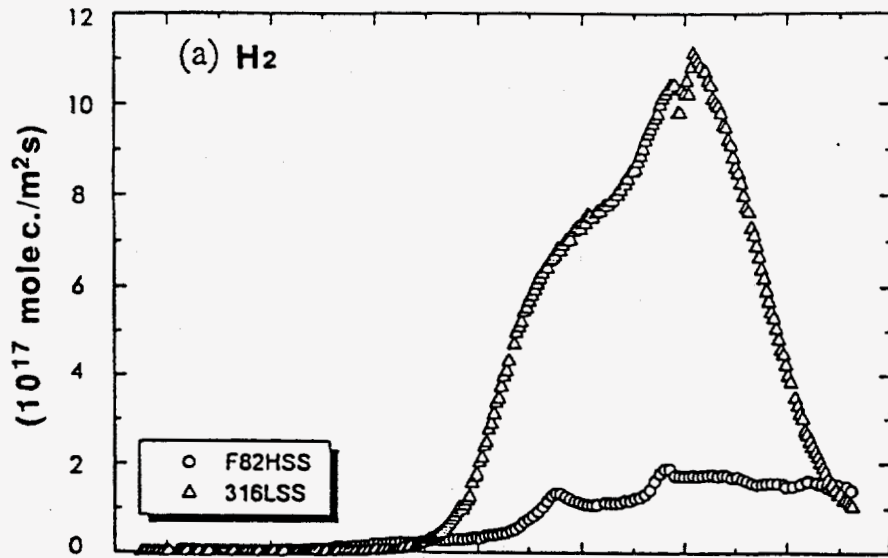
Compared with 316 stainless steels,

- The desorption rate of gas species with oxygen is large
- The sum of desorption volume is comparable
- The activation energy for desorption is small and then, the 2nd peak of desorption rate is shifted to 100-200 K lower temperature





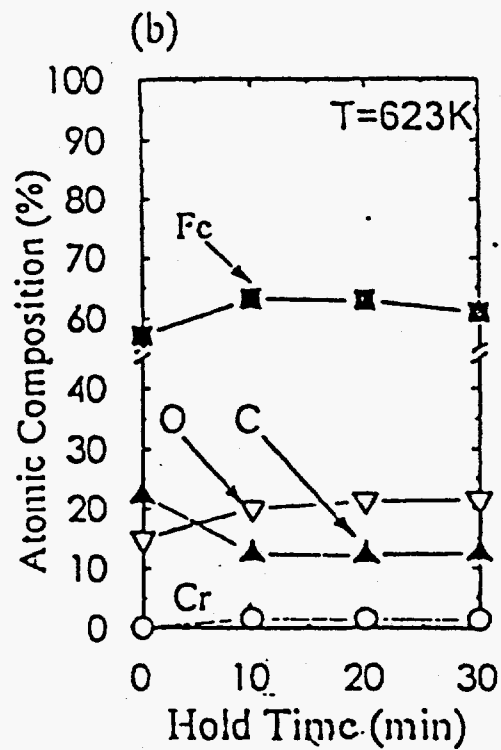
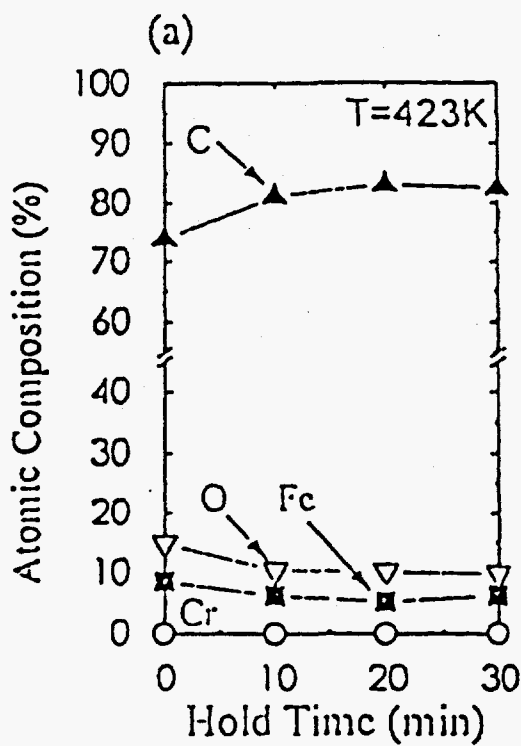
DESORPTION RATE



## (2) Change in Surface Compositions as a Function of Heating Time

- At 423 K,  
oxygen concentration decreases and carbon increase inversely with holding time. The decrease in oxygen is due to desorption of H<sub>2</sub>O.

- At 623 K,  
Iron concentration is about 60 % and carbon decreases rapidly with hold time. This decrease is due to desorption of CO and CO<sub>2</sub>.  
And Chromium segregation is found after 10 min or so.



## Corrosion Tests of F82H

### Subject

Obtain the corrosion property of F82H in high temperature water.

### Test Procedure

In-loop Corrosion Loss Measurement

Material: F82H IEA heat, HT9, Chromized F82H

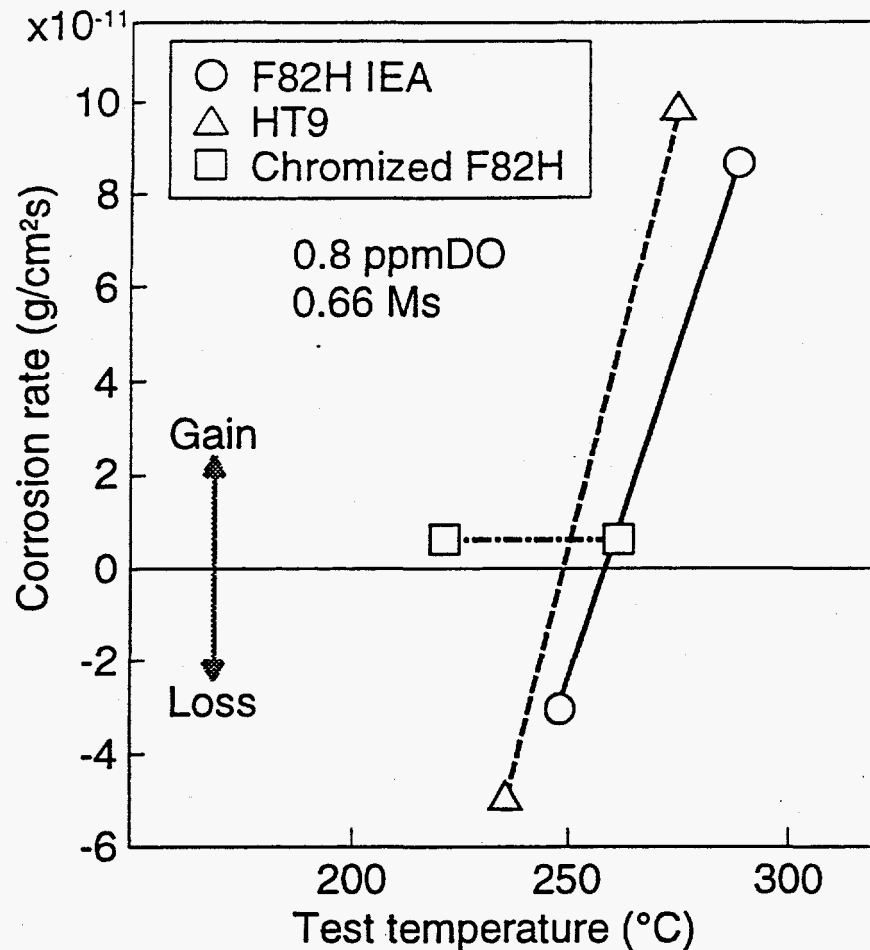
Temperature: 250, 280°C

Dissolved Oxygen: 0.8 ppm

### Result

Alloy	ID	Surface Area (cm <sup>2</sup> )	Volume (cm <sup>3</sup> )	Test Temp. (°C)	DO (ppm)	Weight initial (g)	Test Period (Ms)
F82H	F82H-01	24.61	5.12	248	0.8	40.3733	0.66
F82H	F82H-02	24.62	5.12	288	0.8	40.3944	0.66
HT9	HT9-01	24.55	5.07	235	0.8	39.4329	0.66
HT9	HT9-02	24.68	5.11	274	0.8	39.5460	0.66
Chromized F82H	Crnz-01	24.32	4.99	221	0.8	39.0386	0.66
Chromized F82H	Crnz-02	24.34	5.00	261	0.8	39.0551	0.66
F82H	(after Yamanouchi)			250	0.2		0.9
HT9	(after Yamanouchi)			250	0.2		0.9
18Cr-4W	(after Yamanouchi)			250	0.2		0.9

Alloy	Weight after test (g)	Weight loss			
		(mg)	(g/cm <sup>2</sup> s)	1year (g/cm <sup>2</sup> )	1year mm
F82H	40.3728	-0.5	-3.1E-11	-0.0010	-0.001
F82H	40.3958	1.4	8.63E-11	0.0027	0.003
HT9	39.4321	-0.8	-4.9E-11	-0.0016	-0.002
HT9	39.5476	1.6	9.84E-11	0.0031	0.004
Chromized F82H	39.0387	0.1	6.24E-12	0.0002	0.000
Chromized F82H	39.0552	0.1	6.24E-12	0.0002	0.000
F82H			-1.7E-09	-0.0536	-0.068
HT9			-1.1E-09	-0.0347	-0.044
18Cr-4W			-3E-10	-0.0095	-0.012



#### Temperature Dependence

- More corrosion loss were observed at 250°C than at 280°C in both F82H and HT9, while chromized F82H did not change the weight at both temperature.
- The Oxidized film formed at 280°C is more protective ?

#### Cr content dependence

- F82H showed much weight loss than HT9.

(Yamonouchi, et al., JNM 191-194 (1992), pp.822)

250°C; 0.2 ppmDO; 0.9 Ms in water

F82H:  $-1.7 \times 10^{-9}$  g/cm<sup>2</sup>s

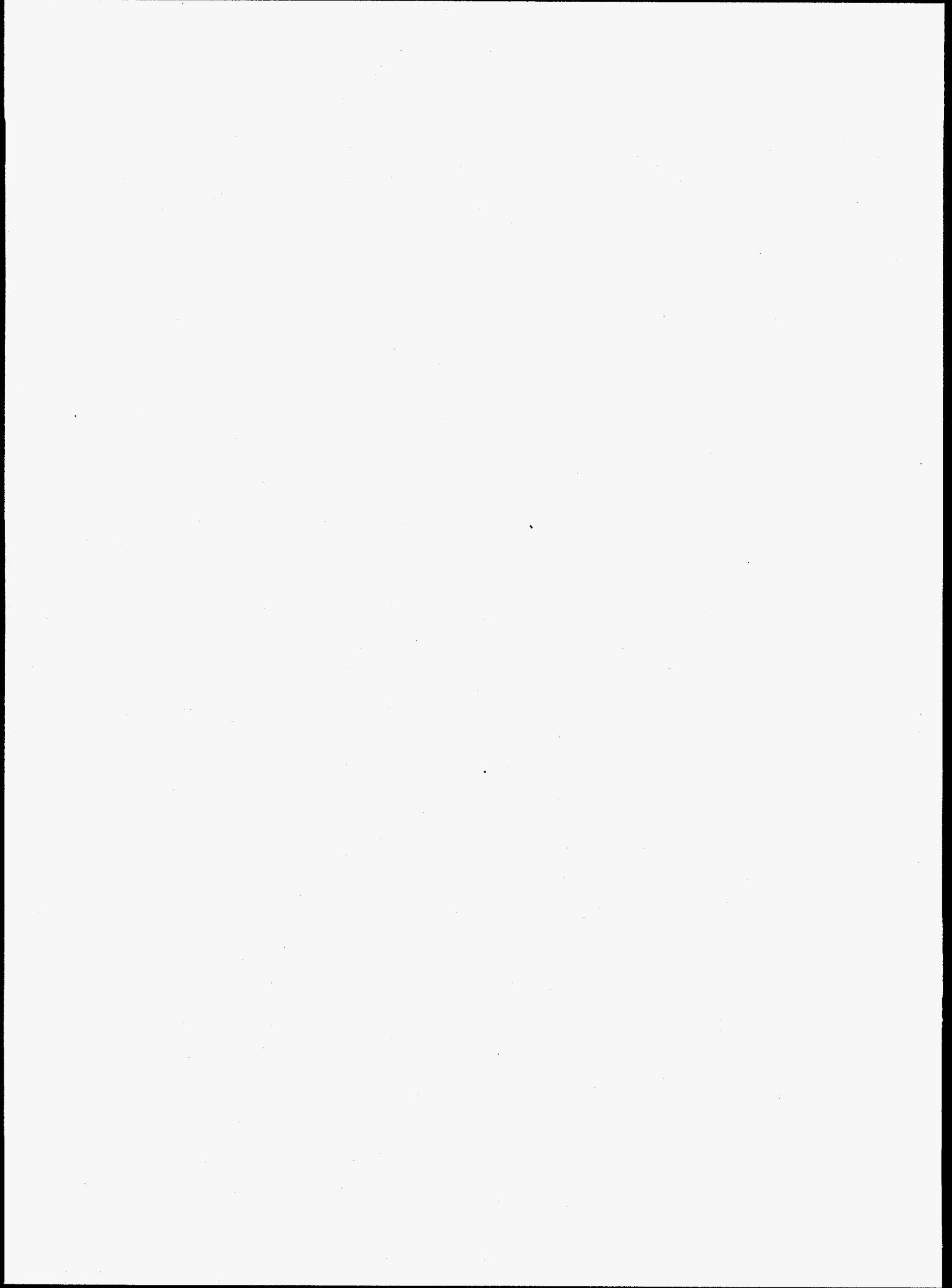
HT9:  $-1.1 \times 10^{-9}$  g/cm<sup>2</sup>s

18Cr-4W:  $-3.0 \times 10^{-10}$  g/cm<sup>2</sup>s

- Corrosion rate of F82H is 50 times higher than present result at 250°C.
- 0.2 ppmDO is not sufficient to form a protective film ?

#### To be confirmed

- Time dependence:
  - Obtain the corrosion rate in a steady state
- Temperature dependence:
  - 200 - 280°C
- Effect of DO:
  - 0.2, 1, 3 ppmDO



# **AN INTEGRATED APPROACH TO ASSESSING THE FRACTURE SAFE MARGINS OF FUSION STRUCTURES**

**G. R. Odette**

**UC Santa Barbara**

**IEA Workshop and Working Group Meeting on Ferritic Steels**

**October 24-25, 1996  
Culham, UK**



# Research Objectives

Integrate measurements/observations with modeling:

*Basic fracture mechanisms* and a fully quantitative micromechanically-based local fracture model

Rigorous methods to: a) *use small specimens to measure the intrinsic fracture resistance of these alloys* ; and b) use the intrinsic properties to predict the load-displacement limits of flawed fusion structures

Microstructure-property-property models of the effect of alloy/processing variables and irradiation on toughness

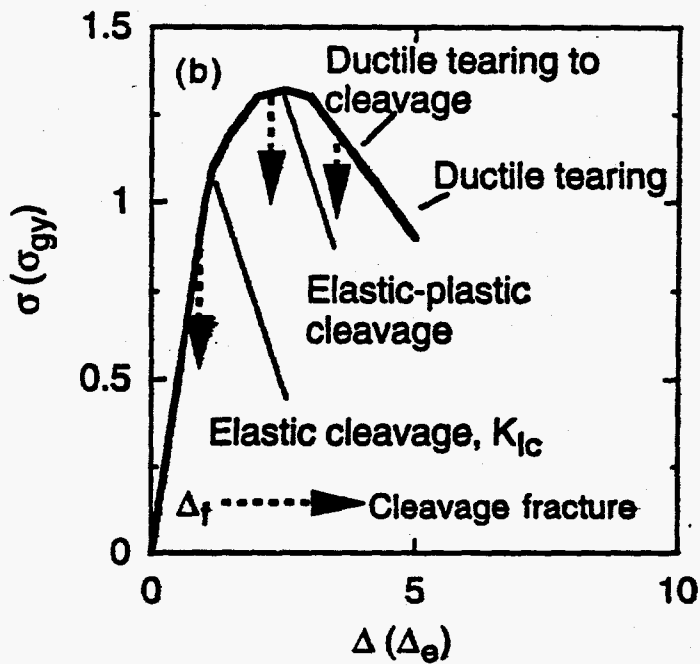
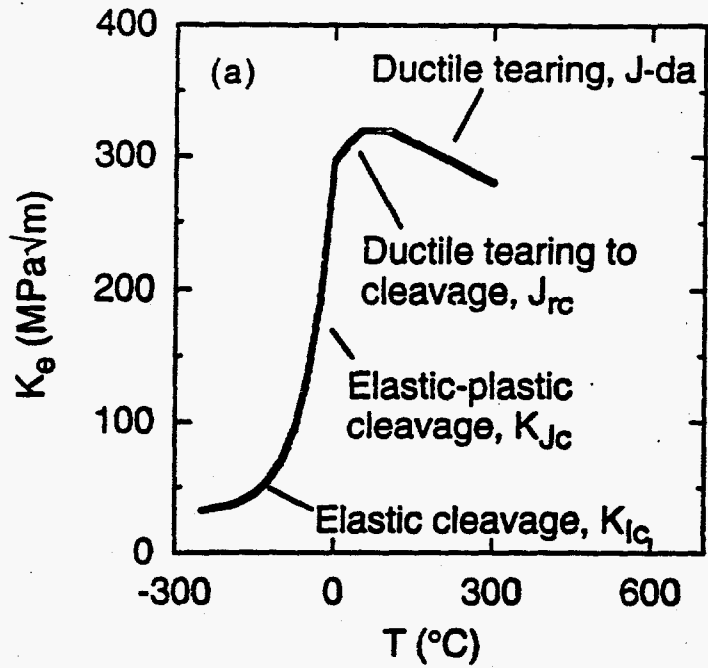
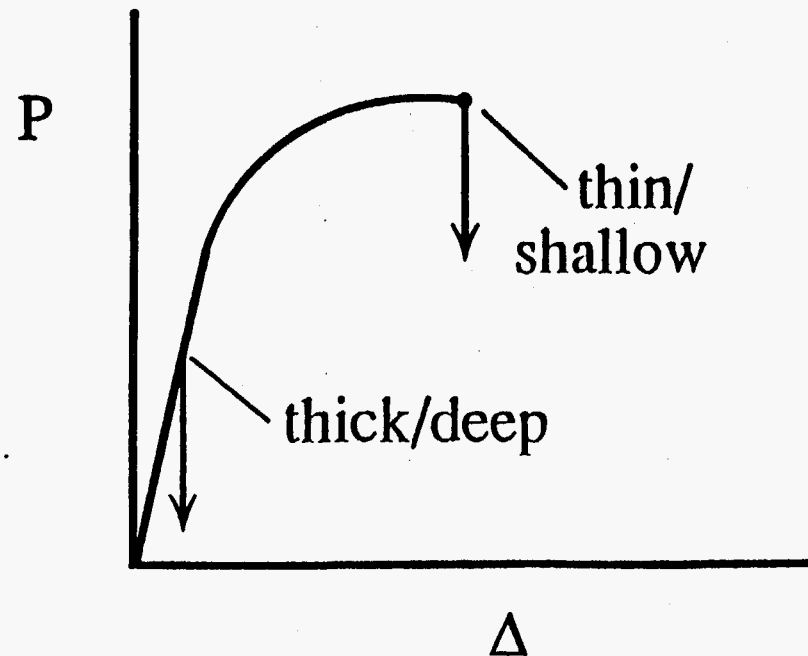
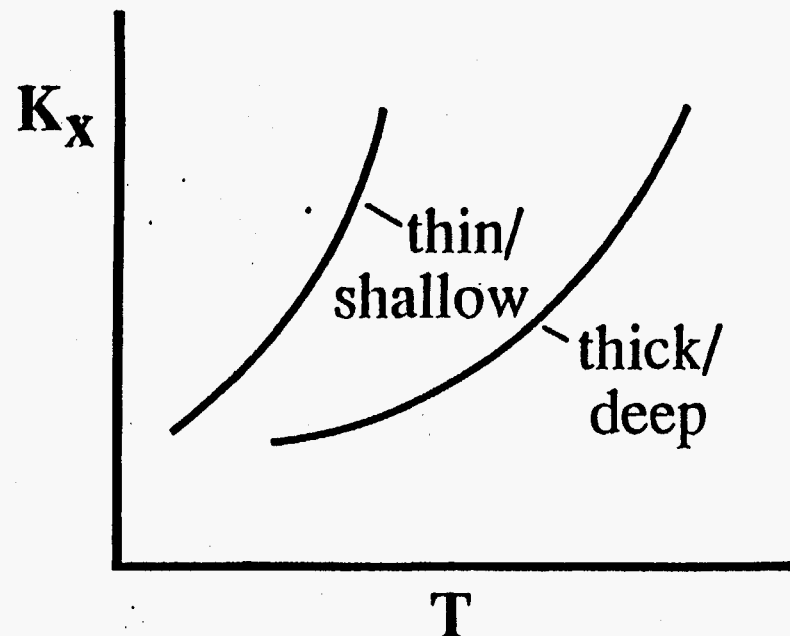


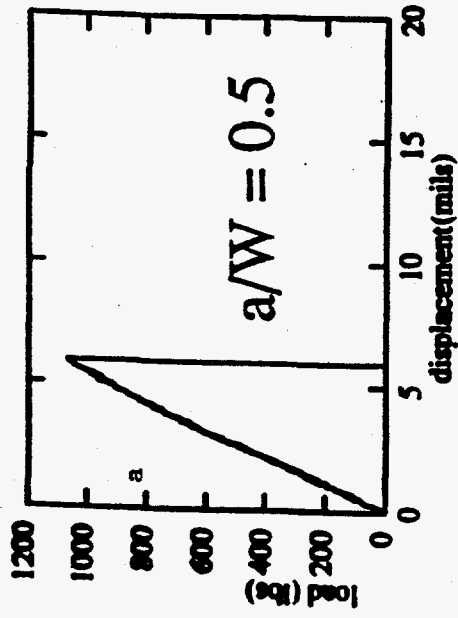
Figure 1 Schematic illustration of a) a  $K_e(T)$  curve showing various fracture regimes; and b) the corresponding macroscopic applied stress-deflection (or strain) curve for a test specimen or structure.

# Role of Size/Geometry

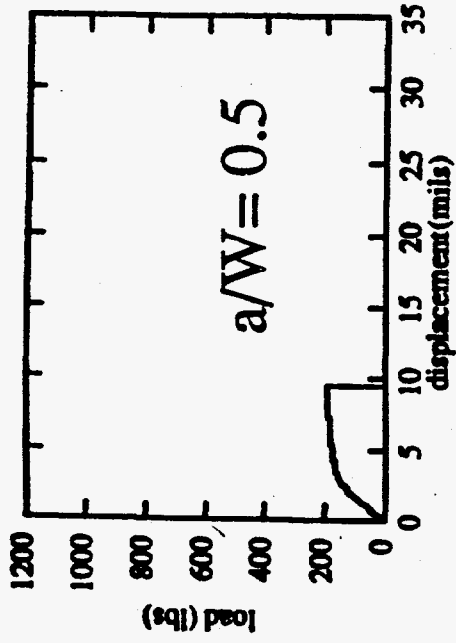
- Same temperature and material conditions ( $K_{Ic/d}$  - cleavage initiation)



PCC



MPCC



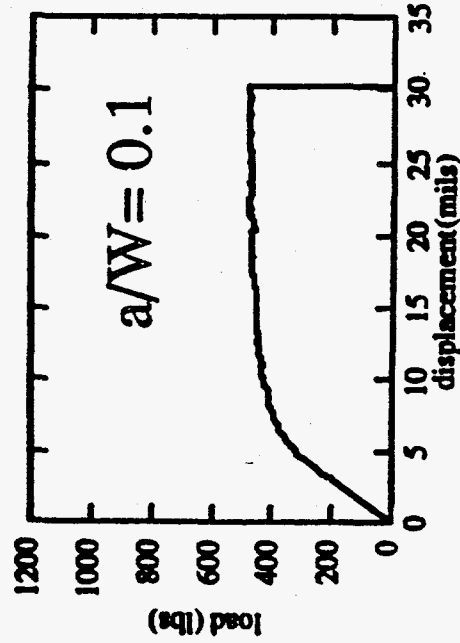
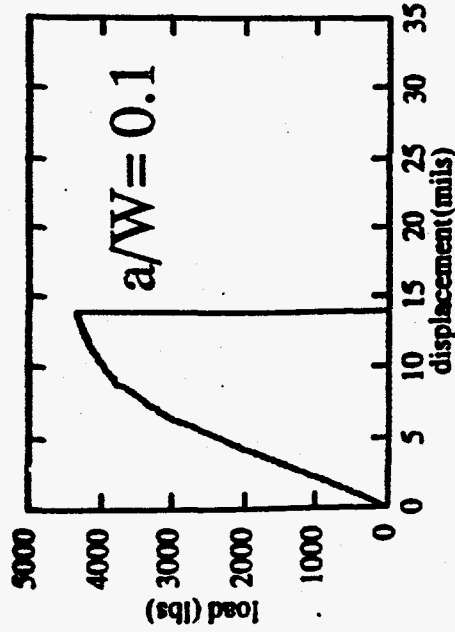
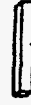
PCC  
 $a/w=0.1$



PCC  
 $a/w=0.5$

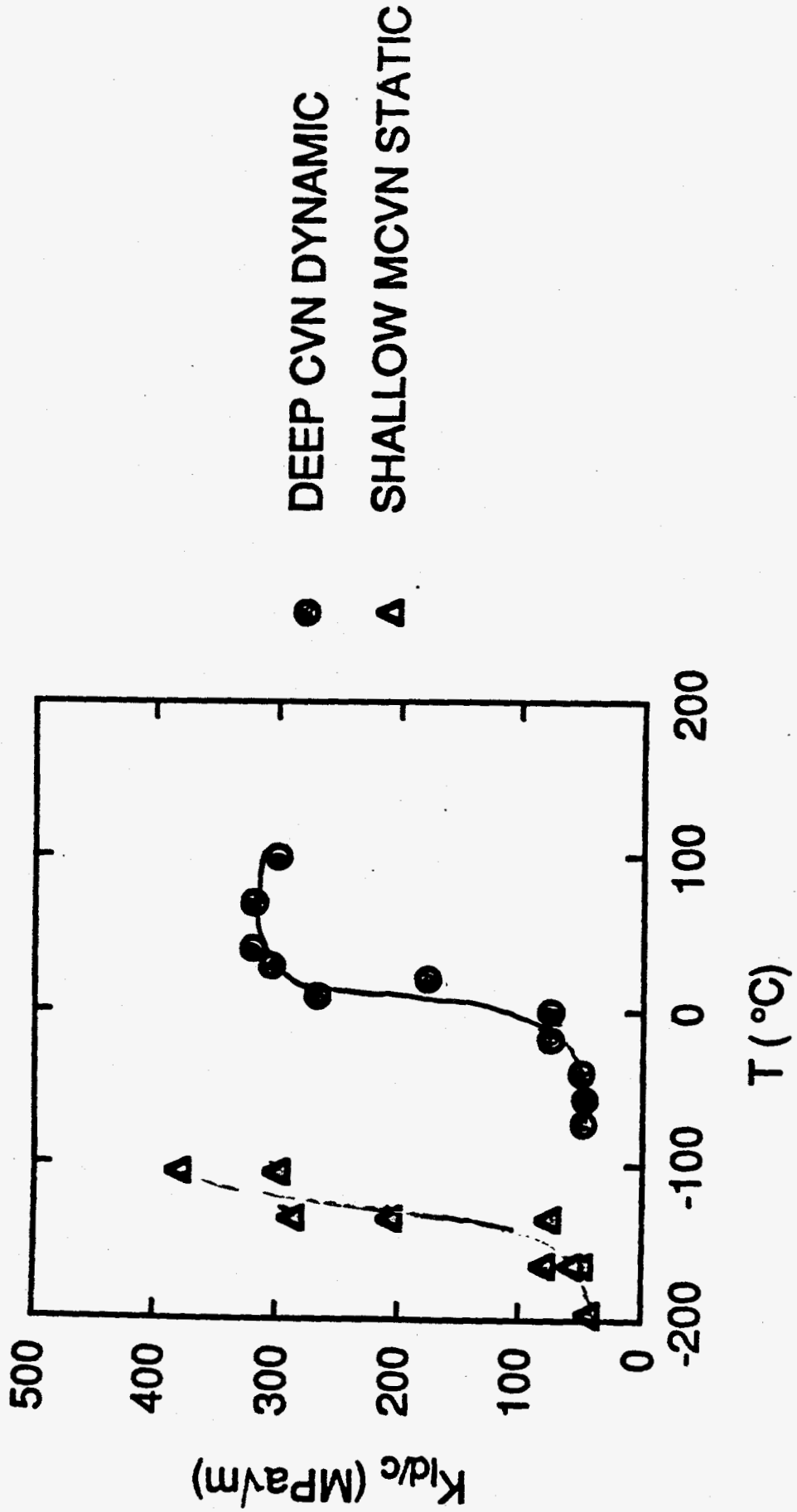


MPCC



F82H @ T = -105°C

# Shallow-Static MCVN vs. Dynamic-Deep CVN



# Unified Master Curves Shift Method

Evidence that pressure vessel steels have a master  $K_{Jc}(T)$  curve shape that is simply shifted in temperature by irradiation (Wallin) --  $K_{Jc}(T)$  from 'small' specimens can be corrected for geometry (statistical) -- subject of a new ASME rule to regulate LWR vessels

TM

$$K_{Jci}(T) = K_{Jcu}(T - \Delta T_{K100} - \text{margin})$$

Need to consider both constraint and statistical effects

Data on F82H suggests there *may* be an approximate master curve for strain rate and geometry. If this also true holds for irradiation

$$K_{Jc(\text{irr}/\dot{\epsilon} / \text{geom})}(T) = K_{Jcu}(T - \Delta T_{\text{irr}} - \Delta T_{\dot{\epsilon}} - \Delta T_{\text{geom}} - \text{margin})$$

**Table 1 -- Values of  $\Delta T$  at 60 MPa $\sqrt{m}$  for Tests on F82H With Various Specimen Configurations and Loading Rates**

Specimen/loading rate		$\Delta T(^{\circ}C)$
Shallow pre-cracked minicharpy	(SPCMC)	-60
Shallow pre-cracked Charpy	(SPCC)	-55
Deeply pre-cracked minicharpy	(PCMC)	-20
Deeply pre-cracked Charpy	(PCC)	0 (reference)
0.6T bend bar (about 3x Charpy size)	(0.6TBB)	+10
Dynamic deeply pre-cracked minicharpy	(DPCMC)	+50
Dynamic deeply pre-cracked Charpy	(DPCC)	+80

# Unified Master Curves Shift Method

Evidence that pressure vessel steels have a master  $K_{Jc}(T)$  curve shape that is simply shifted in temperature by irradiation (Wallin) --  $K_{Jc}(T)$  from 'small' specimens can be corrected for geometry (statistical) -- subject of a new ASME rule to regulate LWR vessels

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$$K_{Jc(\text{irr}/\dot{\epsilon} / \text{geom})}(T) = K_{Jcu}(T - \Delta T_{\text{irr}} - \Delta T_{\dot{\epsilon}} - \Delta T_{\text{geom}} - \text{margin})$$



# Master Curves

For low alloy RPV steels

$$K_e(T) = 30 + 30 \exp[A(T' - T_0)] \text{ (MPa}\sqrt{\text{m)}})$$

where  $A = 0.019$  and  $T'$  is an adjusted temperature scale to account for irradiation, loading rate,...

$$T' = T - \Delta T_0$$

and  $T_0$  is a reference temperature for a particular steel/test /toughness index: F82H/static deeply pre-cracked Charpy/60 MPa $\sqrt{\text{m}}$ ,  $T_0 = -115^\circ\text{C}$

RPV form works well for deep cracks, larger specimens, static rates; for shallow cracks and/or small specimens and/or dynamic rates  $A = 0.038$  provides a 'better' fit

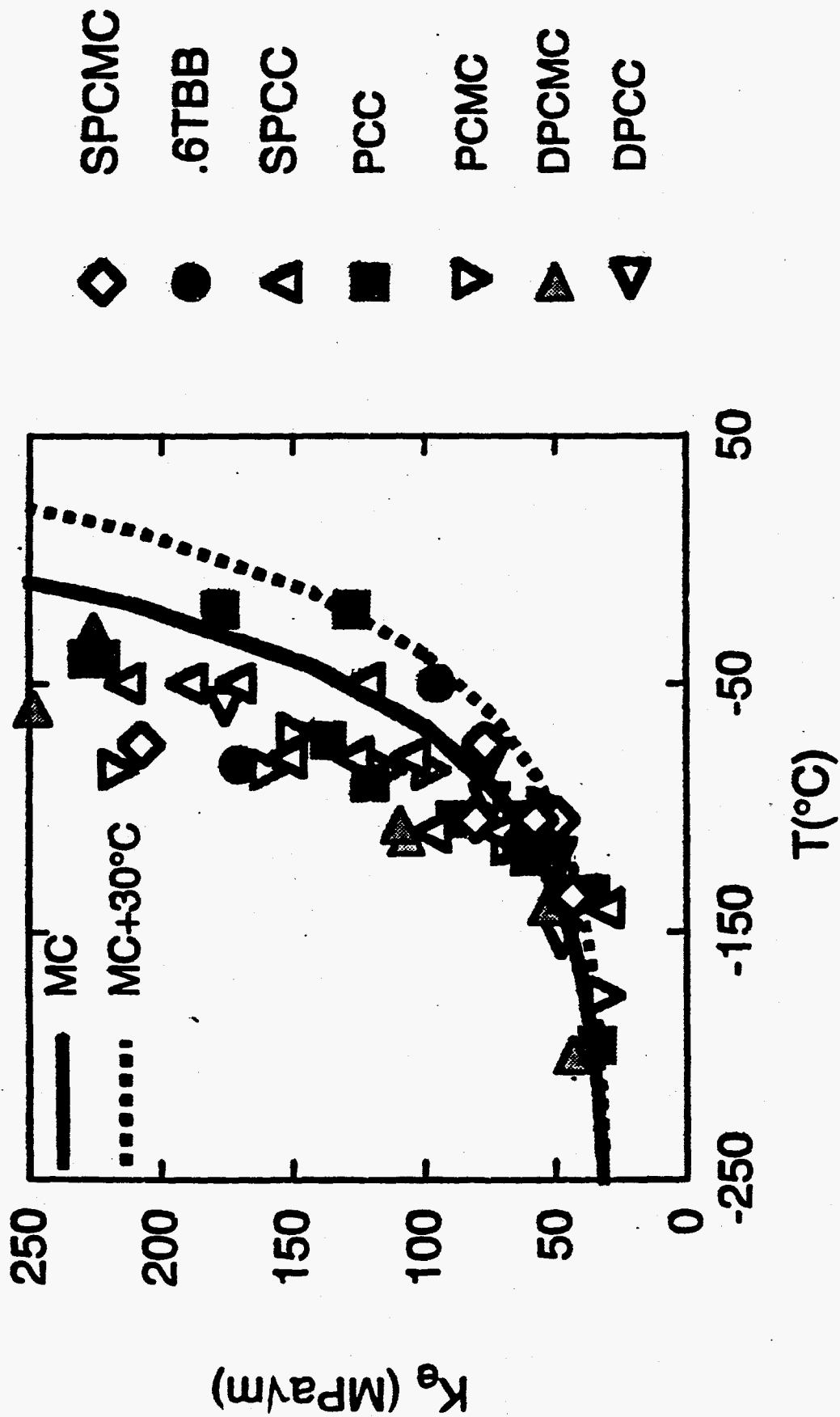


Figure 3 The adjusted  $K_e(T)$  curves compared to the RPV MC and MC + 30°C bound.

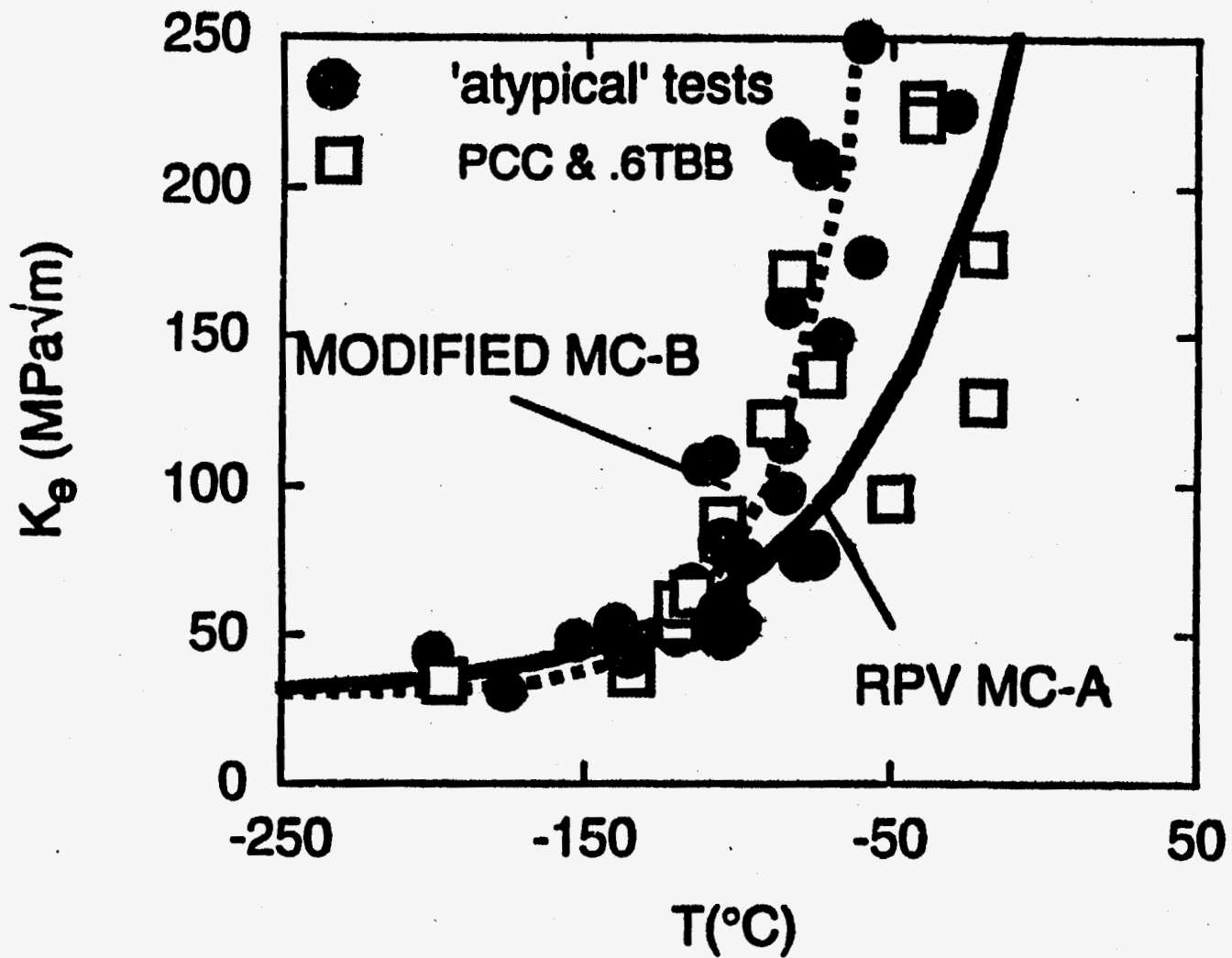
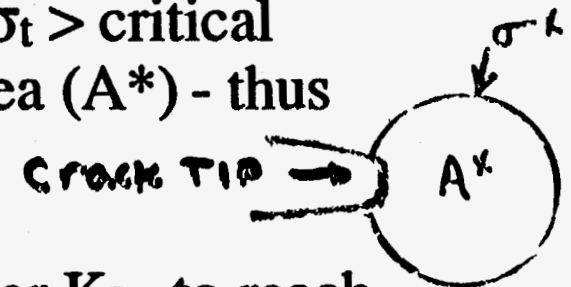


Figure 4 The  $K_e(T)$  data for atypical tests (circles) versus PCC and 0.6TBB data (squares) along with the RPV MC (A) and modified MC (B).

# Micromechanics (greatly simplified)

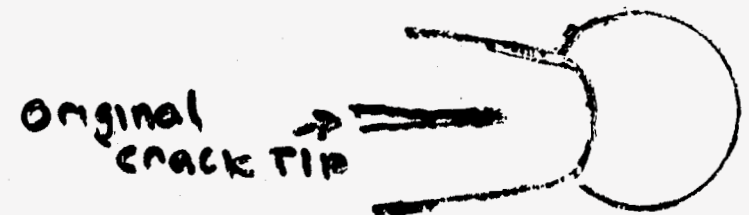
The high near tip tensile stresses ( $\sigma_t$ ) for deep/large small scale yielding (ssy) cracks peak at a small multiple ( $M_{ssy} \approx 3-5$ ) of the yield stress ( $\sigma_y$ ) and the peak stressed area varies as  $K_I^4$

Quasicleavage occurs at  $K_{Ic}$  when a contour of  $\sigma_t >$  critical cleavage fracture stress  $\sigma^*$  encloses a critical area ( $A^*$ ) - thus  $\sigma^*/A^*$  are local measures of toughness



Shallow/small cracks  $M < M_{ssy}$  requiring a higher  $K_{Ic}$  to reach the same local  $\sigma^*/A^*$ . If  $M\sigma_y < \sigma^*$  fracture occurs by ductile micro-void coalescence

Similar concepts for notched tests



'Explains' the DBT and effect of size, rate and irradiation

# Equivalent Yield Stress Model

If  $\sigma^*/A^* \neq f(T, \phi t, \dot{\epsilon})$ , a simple way to model  $\Delta T$  is to define temperatures at equal reference yield stress conditions

The reference condition is at the highest temperature ( $T_r$ ) of elastic cleavage fracture --  $K_{Jc} \approx 60 \text{ MPa}\sqrt{\text{m}}$  or  $E_{cvn} \approx 10 \text{ J}$

$\Delta T_r$  can be modeled from  $\sigma_y(\dot{\epsilon}, T, \phi t)$

## $\Delta T - \Delta \sigma_y$ Models

Reasonable approximations (RPV steels) may include

$$\sigma(\epsilon) = \sigma_y(\epsilon/\epsilon_y)^n \quad \text{and } n \approx C/\sigma_y$$

$$\sigma_{yi}(\dot{\epsilon}, T) = \sigma_{yu}(\dot{\epsilon}, T) + \Delta\sigma_{yi}$$

$$T(\dot{\epsilon}) = T_{\dot{\epsilon}_r} [1 + C \ln(\dot{\epsilon}_r/\dot{\epsilon})]$$

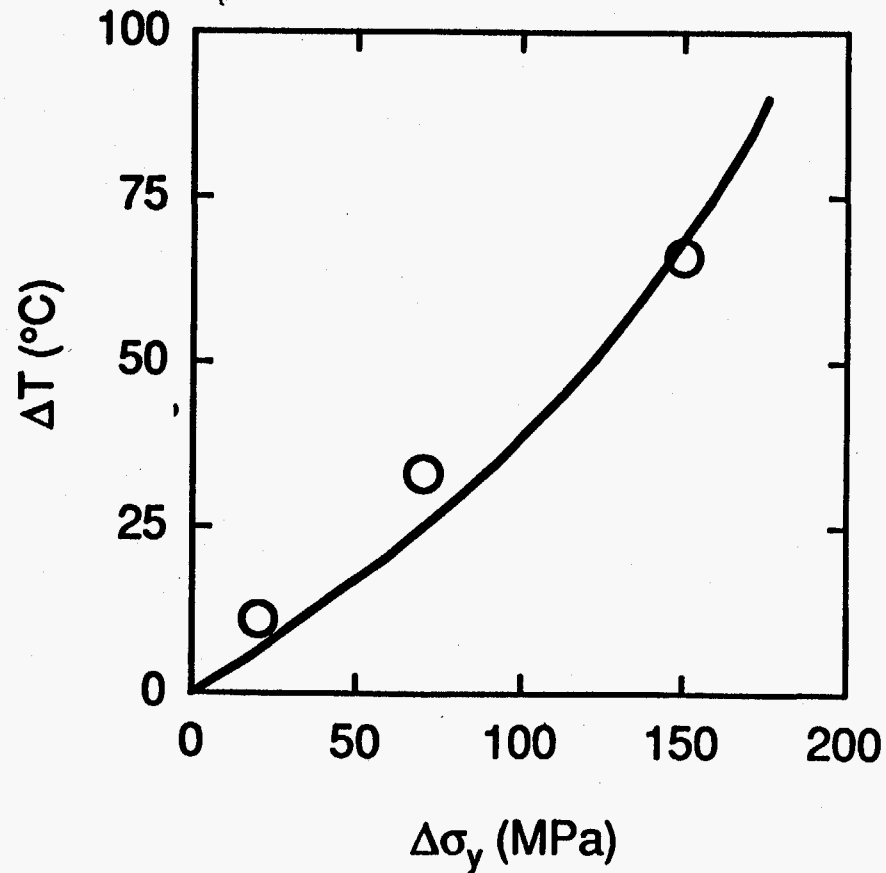
$$\Delta T_r = f(T_r, \Delta\sigma_{yi}, \dot{\epsilon}_r/\dot{\epsilon})$$

**Table 2 - Predicted vs. Measured Static to Dynamic  $\Delta T_d$**

<b>Specimen</b>	<b><math>T_{os}</math> (°C)</b>	<b><math>\Delta T_{dp}</math> (°C)</b>	<b><math>\Delta T_{dm}</math> (°C)</b>
PCC	-115	75	80
PCMC	-135	65	70

## Irradiation Embrittlement of F82H

Compare equivalent yield stress model predictions to data of M. Reith et. al. (Karlsruhe) on  $\Delta\sigma_{yd}$  (dynamic @ 100°C) and MCVN shifts





**Table 3 - Predicted vs. Measured  $\Delta T_i$  for MCVN Tests on 9 Cr Steels**

<b>Spec./Prop.</b>	<b><math>T_{ou}</math>(°C)</b>	<b><math>\Delta\sigma_i</math> (MPa)</b>	<b><math>\Delta T_{ip}</math>(°C)</b>	<b><math>\Delta T_{im}</math>(°C)</b>
<b>9Cr-2VW</b>	<b>-120</b>	<b>161</b>	<b>68</b>	<b>68</b>
<b>9Cr-2VW-Ta</b>	<b>-150</b>	<b>125</b>	<b>36</b>	<b>11</b>

## Geometry Effects on $K_e(T)$

FEM ( $\sigma^*/A^*$ ) approximately predicts PCC vs PCMC for both deep and shallow cracks - but,  $(\sigma^*/A^*)_{\text{shallow}} \neq (\sigma^*/A^*)_{\text{deep}}$

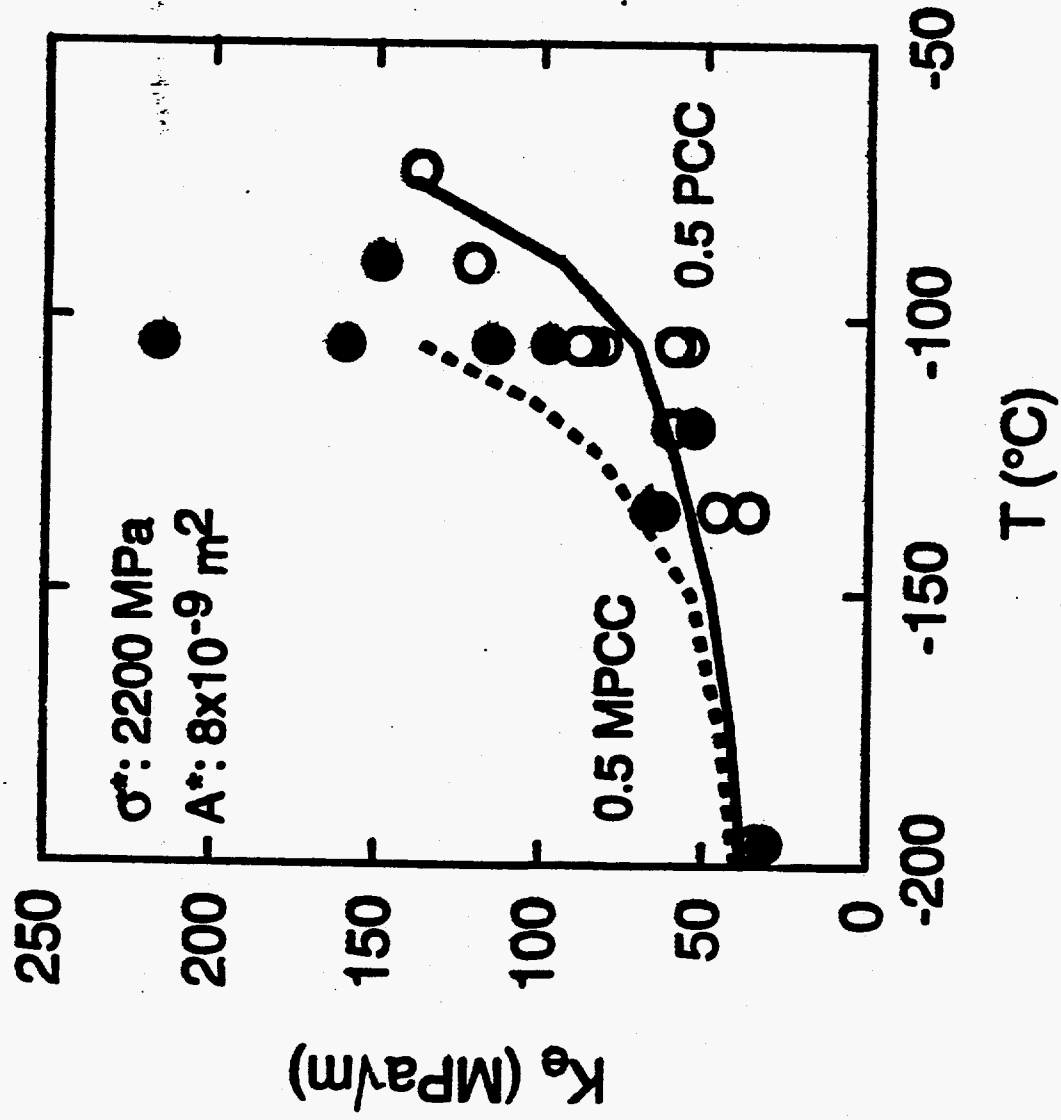


Figure 5  $K_e(T)$  curves predicted by the  $\sigma^*/A^*$  model compared to data for PCMC and PCC specimens.

# Irradiation Effects on $K_e(T)$

No reliable F82H data -- use RPV steel surrogate - FEM ( $\sigma^*/A^*$ ) or EYSM model of hardening effects approximately predicts shifts at  $60 \text{ MPa}\sqrt{\text{m}}$  for MPCC (1/3) and 1CT data but shape change is not observed

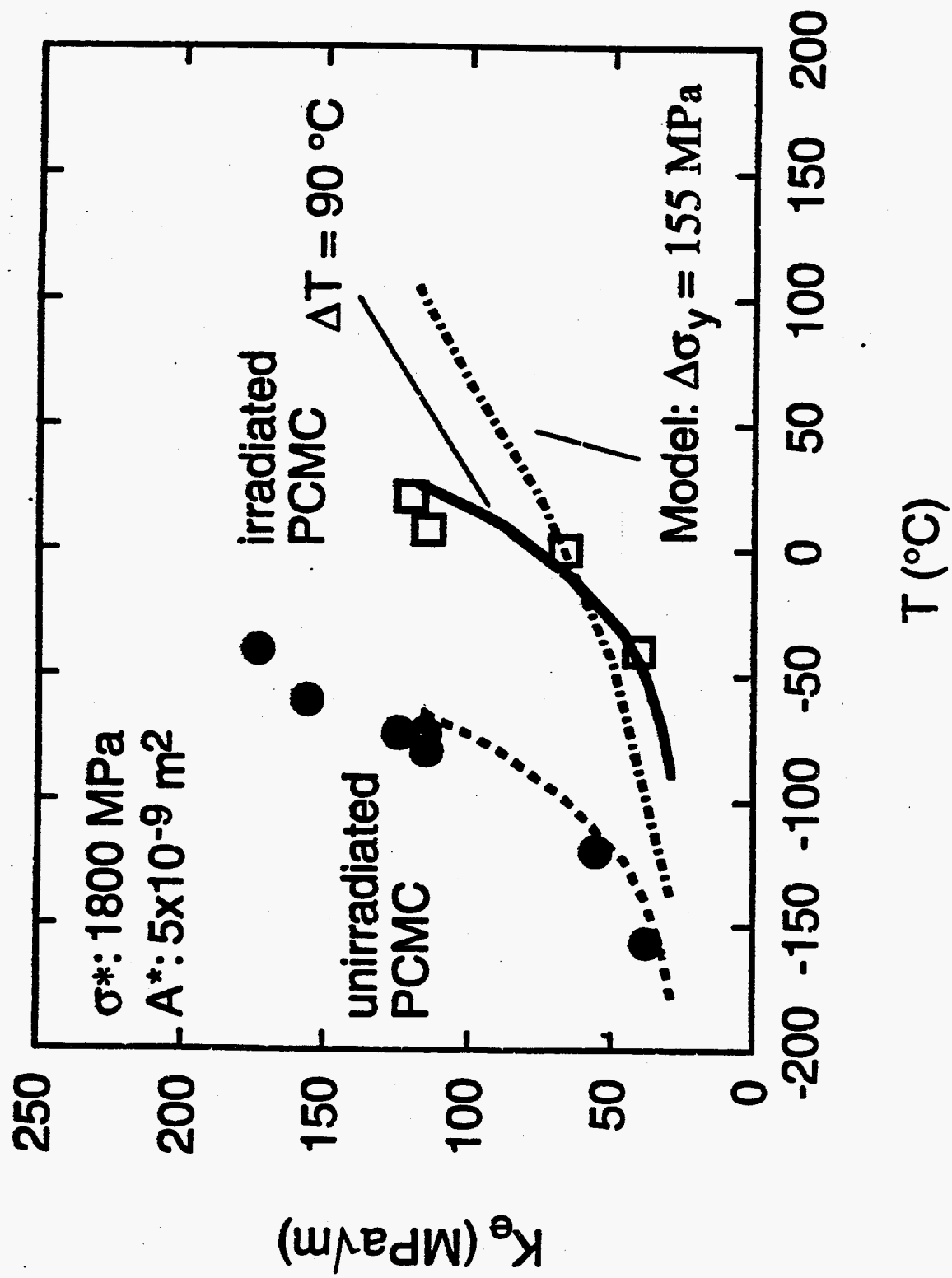


Figure 6 Predicted versus measured shifts in the PCMC  $K_e(T)$  curve for an A533B RPV steel due to irradiation resulting in  $\Delta \sigma_i$  of 155 MPa.

# Engineering Example

Assume a small  $a = 0.3$  cm deep part-through crack in a thin  $W = 1$  cm with a 0.9 cm length at the surface subject to a disruption stress peaking at about 1 ms

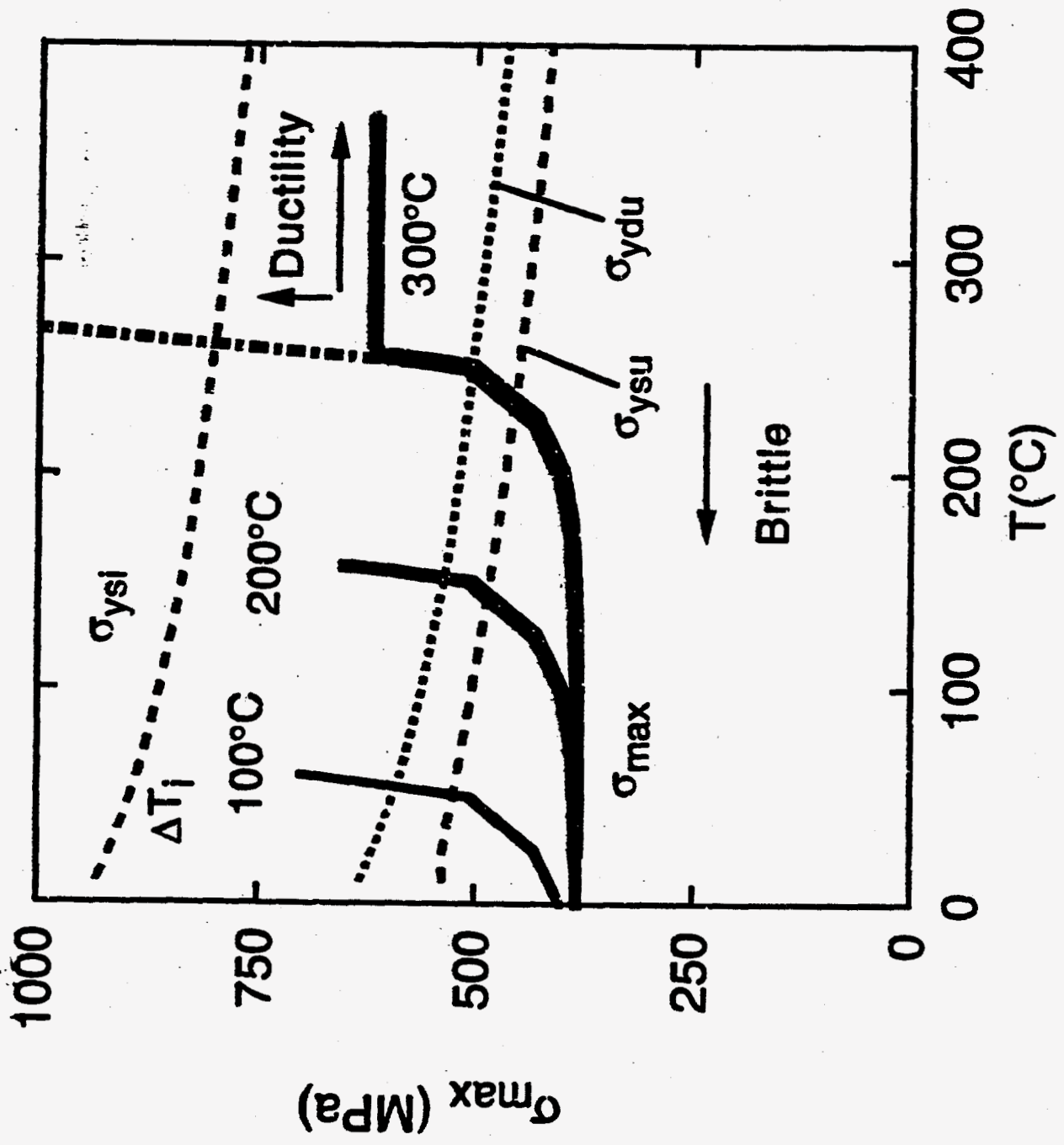
Use MC-B and estimate loads with LEFM method after adjusting for size (s), loading rate (d), 'shallow' crack depth ( $a/W$ ); irradiation (i) and a safety margin (m)

For  $\Delta T_i = 100, 200$  and  $300^\circ\text{C}$  the loads below the rapid upswing would be limited to about 200 MPa assuming a stress safety factor of 2. At higher temperatures, if fracture occurred at all, it would be ductile (requiring a detailed FEM analysis). However, this would be associated with overloads relative to the unirradiated yield stress

The 'allowed' stress-temperature provides operational flexibility assuming load management - like an operating P-T curve for a PWR

**Table 4 -  $\Delta T$  Values Used in the MC- $\Delta T$  Application Example**

Adjustment	Temperature/Shift( $^{\circ}$ C)
$T_o$	-115
$\Delta T_s$	+ 10
$\Delta T_d$	+ 80
$\Delta T_{a/w}$	- 20
$\Delta T_i$	+100, 200, 300
$\Delta T_m$	+ 60





# Example (Only) Safety Scenerio

Startup in brittle regime:

$$\sigma_p + \sigma_{th} < 0.33 \sigma_{max}$$

Operating in the ductile regime:

$$2\sigma_p + \sigma_{th} < 0.9\sigma_{ysu}$$

'Rare' transient

$$\sigma_p + \sigma_{th} < 0.9\sigma_{ysu}$$

# Summary and Conclusions

F82H undergoes 'normal' stress controlled cleavage with toughness increasing with temperature  $K(T)$

May be a master  $K(T)$  curve that can be adjusted for strain rate, irradiation, size and geometry

Irradiation (up to a point) and strain rate shifts in the maximum temperature of elastic cleavage fracture can be estimated at an equivalent yield stress

Size effects (up to a point) can also be treated based on a local  $\sigma^*/A^*$  + FEM/constitutive law approach (analogous to RPV steels)

Unresolved issues regarding the shape of  $K(T)$  curve and shallow cracks

# Summary and Conclusions

The reference toughness-multiple shift method may offer a simple and practical means of evaluating irradiation effects on the fracture resistance of flawed fusion structures in realistic geometries and subject to realistic loading conditions -- including high rates and considering system compliance

While the method is quantitative and consistent with a irradiated data base primarily generated using small specimens (and is certainly far superior to any Charpy based approach) it must be experimentally verified

While it may not be practically necessary to go beyond such empirical verification, local fracture mechanics method of measurement and analysis offers and even great promise of a unified and fundamentally based approach to assessing the integrity margins of flawed fusion structures

# FM steel irradiation programme

Presented at IEA FM meeting

at JET Culham

October 24, 1996

Marc G. Horsten

ECN Nuclear Energy

# Outline presentation

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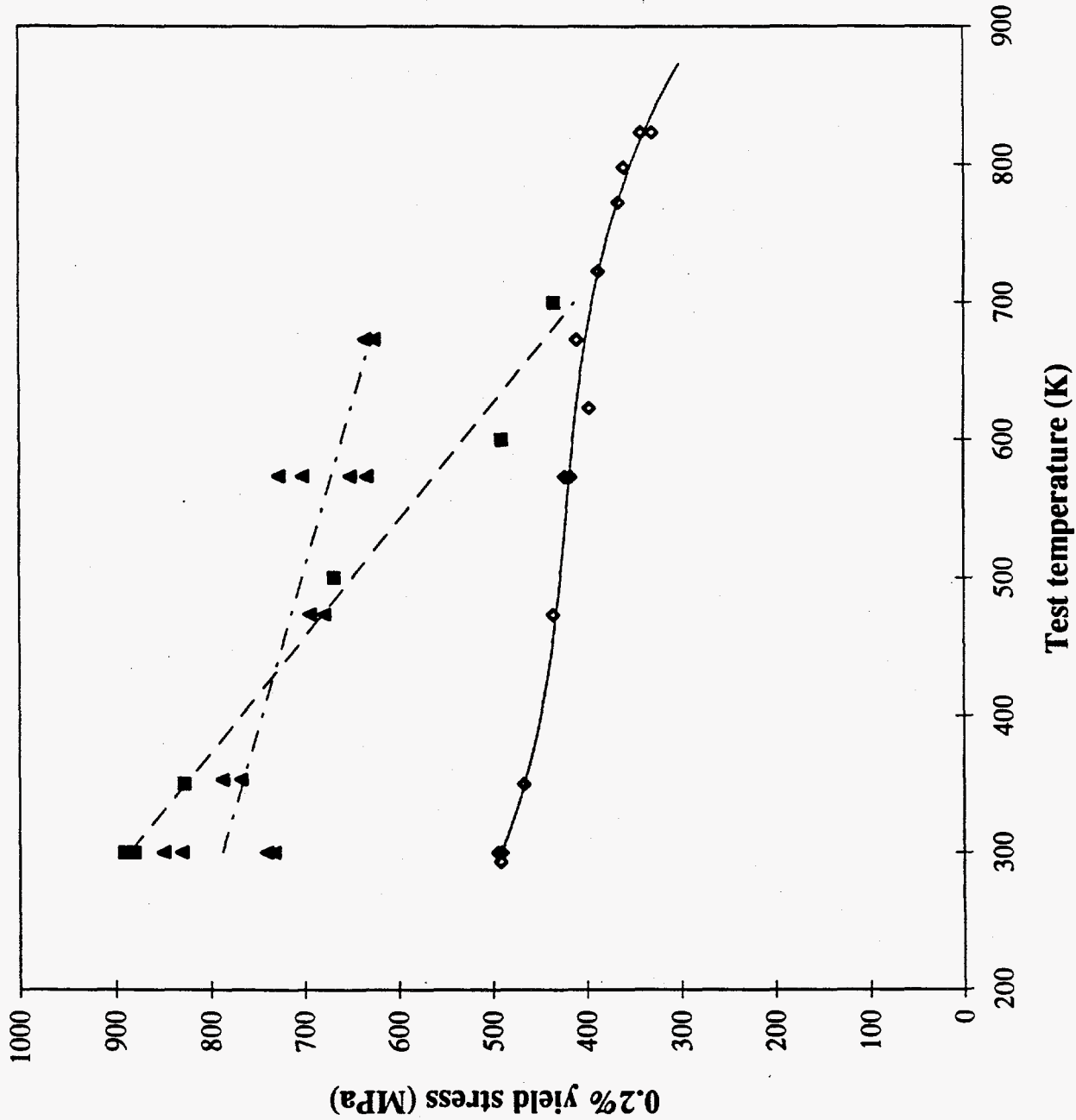
- ◆ Aim of irradiation experiments
- ◆ Mod. 9%Cr, HT-9, and MANET results
- ◆ LA steel reference results
- ◆ Irradiation programme
- ◆ Joint work
- ◆ Conclusions

# Aim of irradiation experiments

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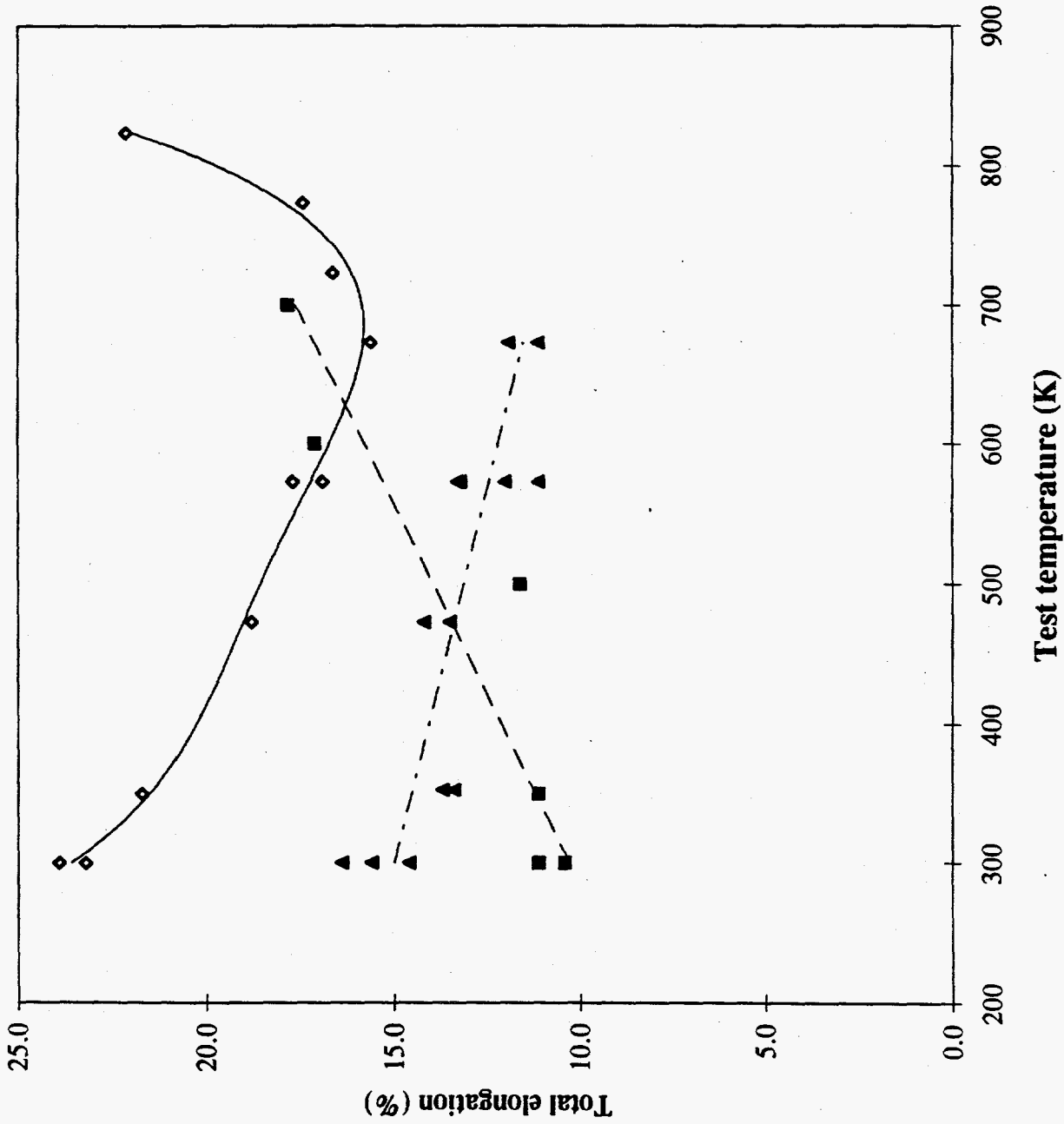
- ◆ Low temperature (<573 K) irradiation embrittlement of 8-12%Cr steels

### Mod 9Cr-F yield stress



◆ Reference  
■ 1.5 dpa at 350 K (SIWAS-5)  
▲ 2.5 dpa at 573 K (ILAS-2)

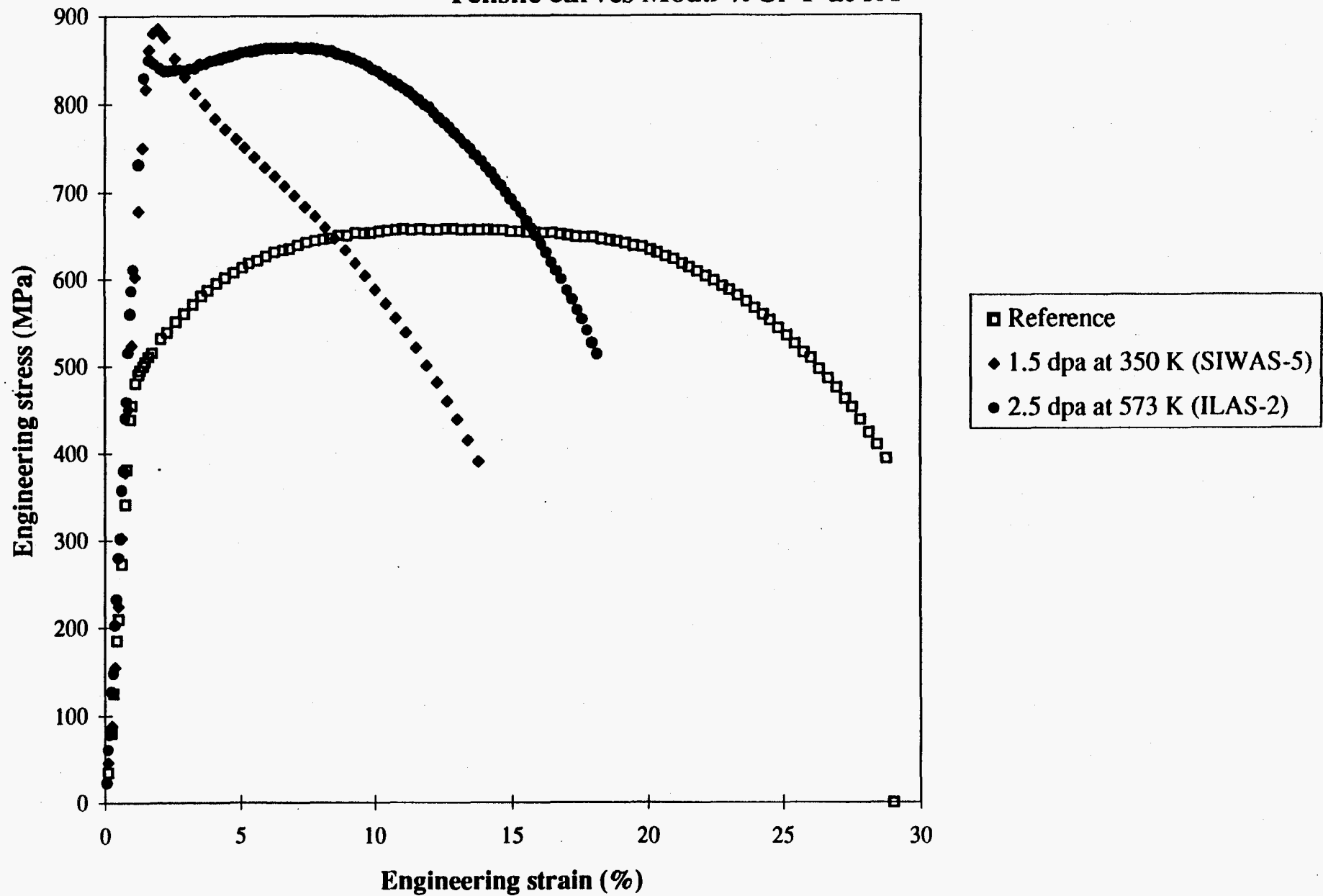
### Mod 9Cr-F ductility



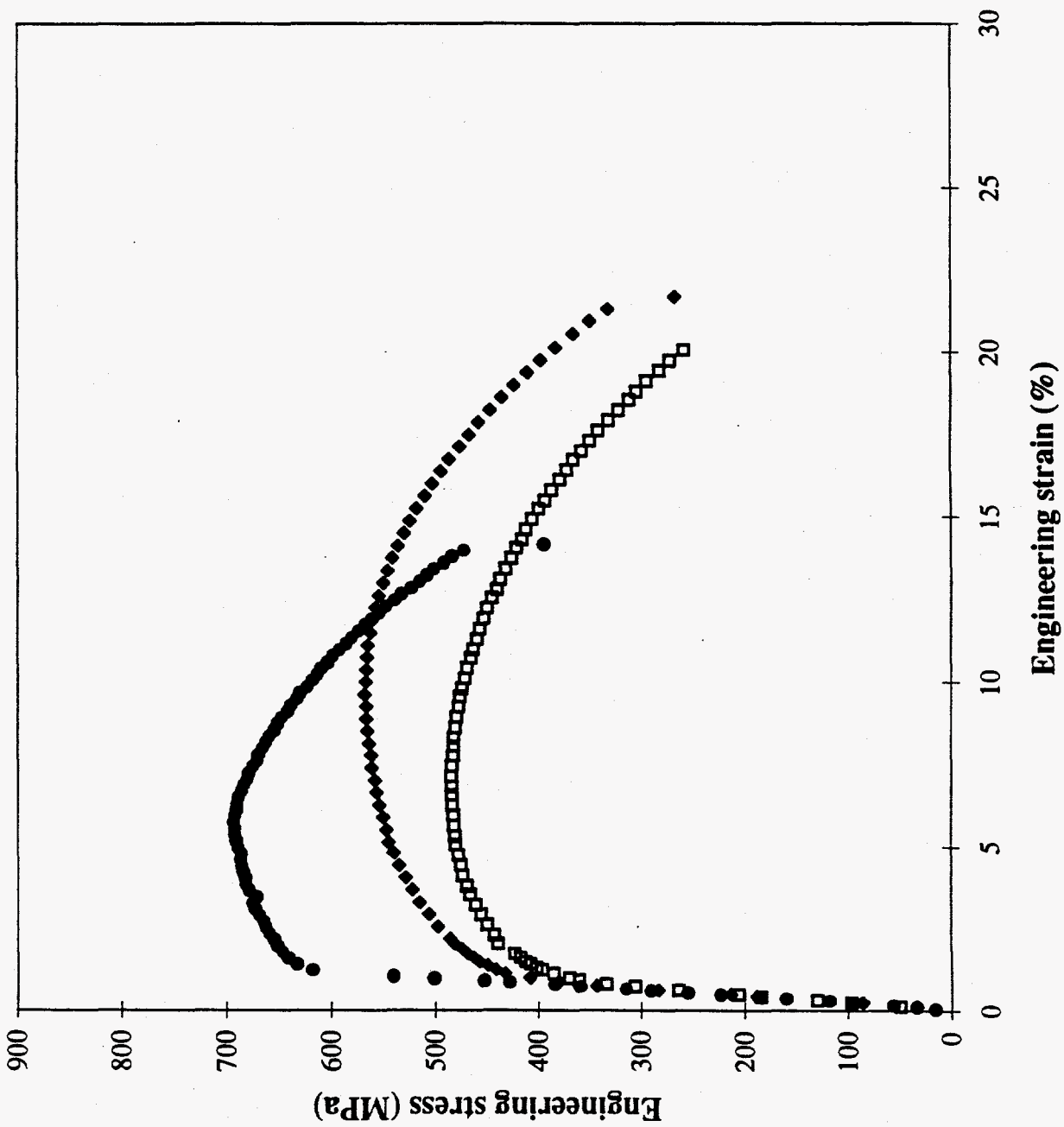
◆ Reference  
■ 1.5 dpa at 350 K (SIWAS-5)  
▲ 2.5 dpa at 573 K (ILAS-2)



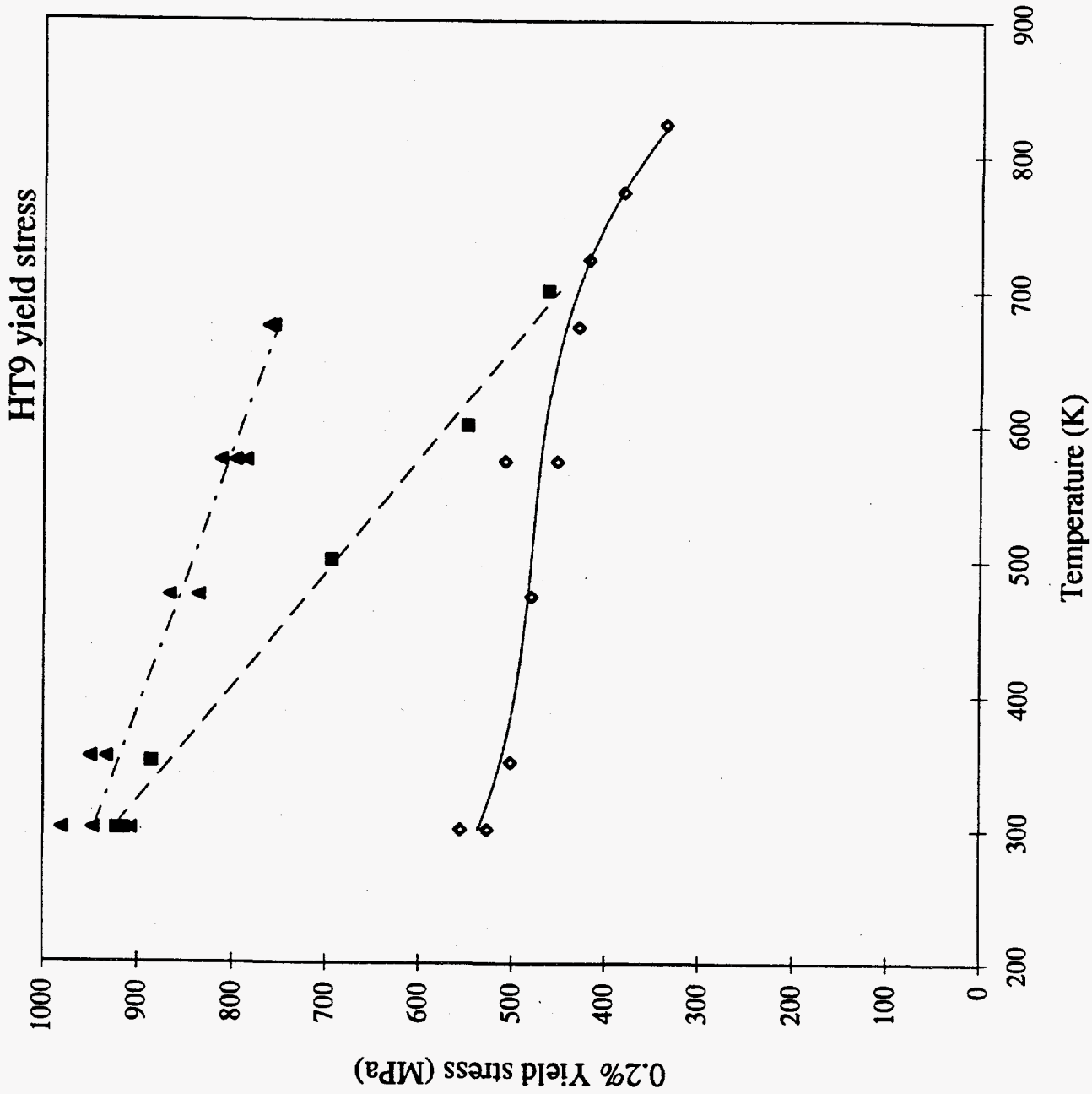
Tensile curves Mod.9%Cr-F at RT



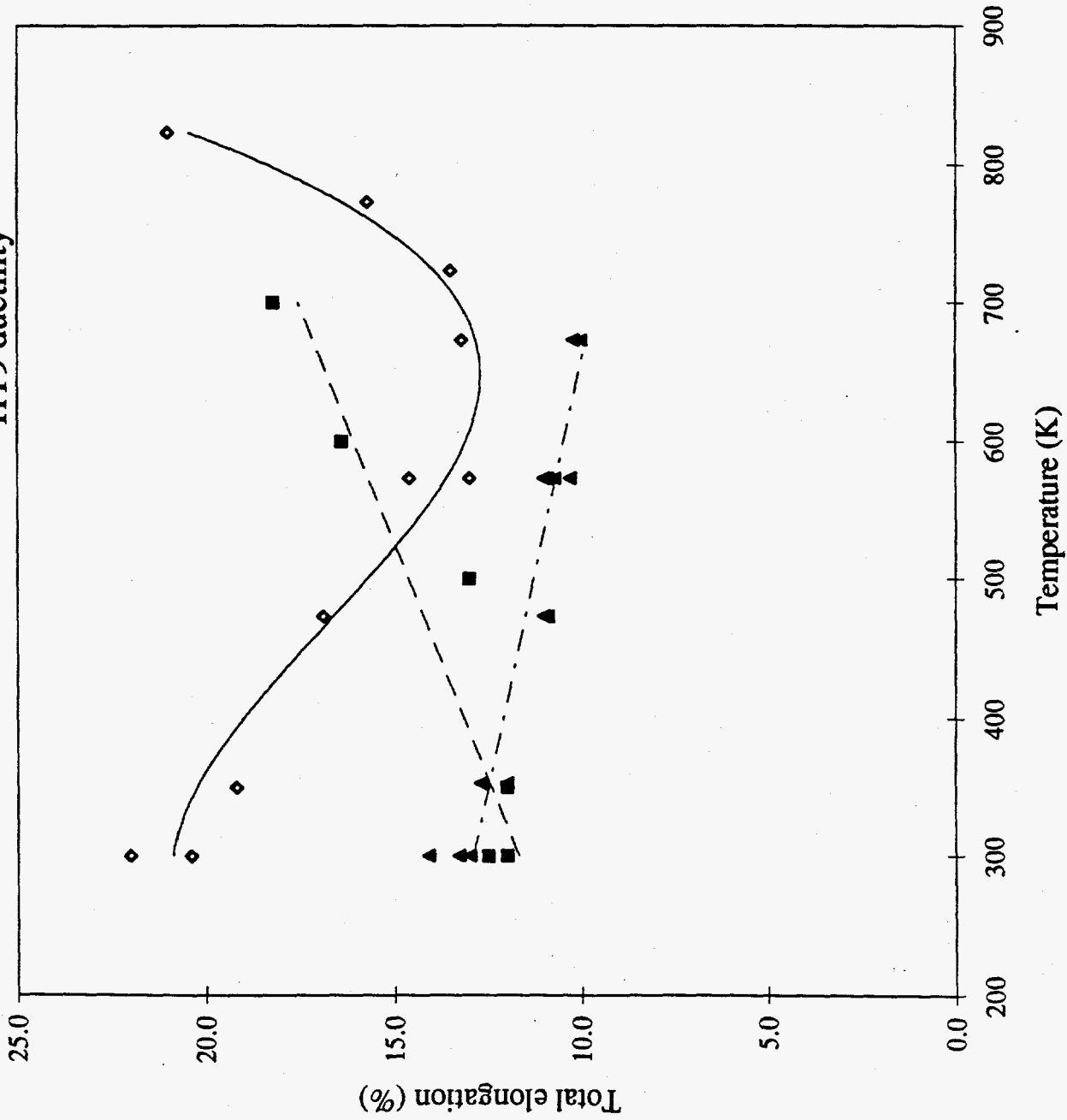
### Tensile curves Mod.9%Cr-F at 673-723 K



□ Reference  
◆ 1.5 dpa at 350 K (SIWAS-5)  
● 2.5 dpa at 573 K (ILAS-2)



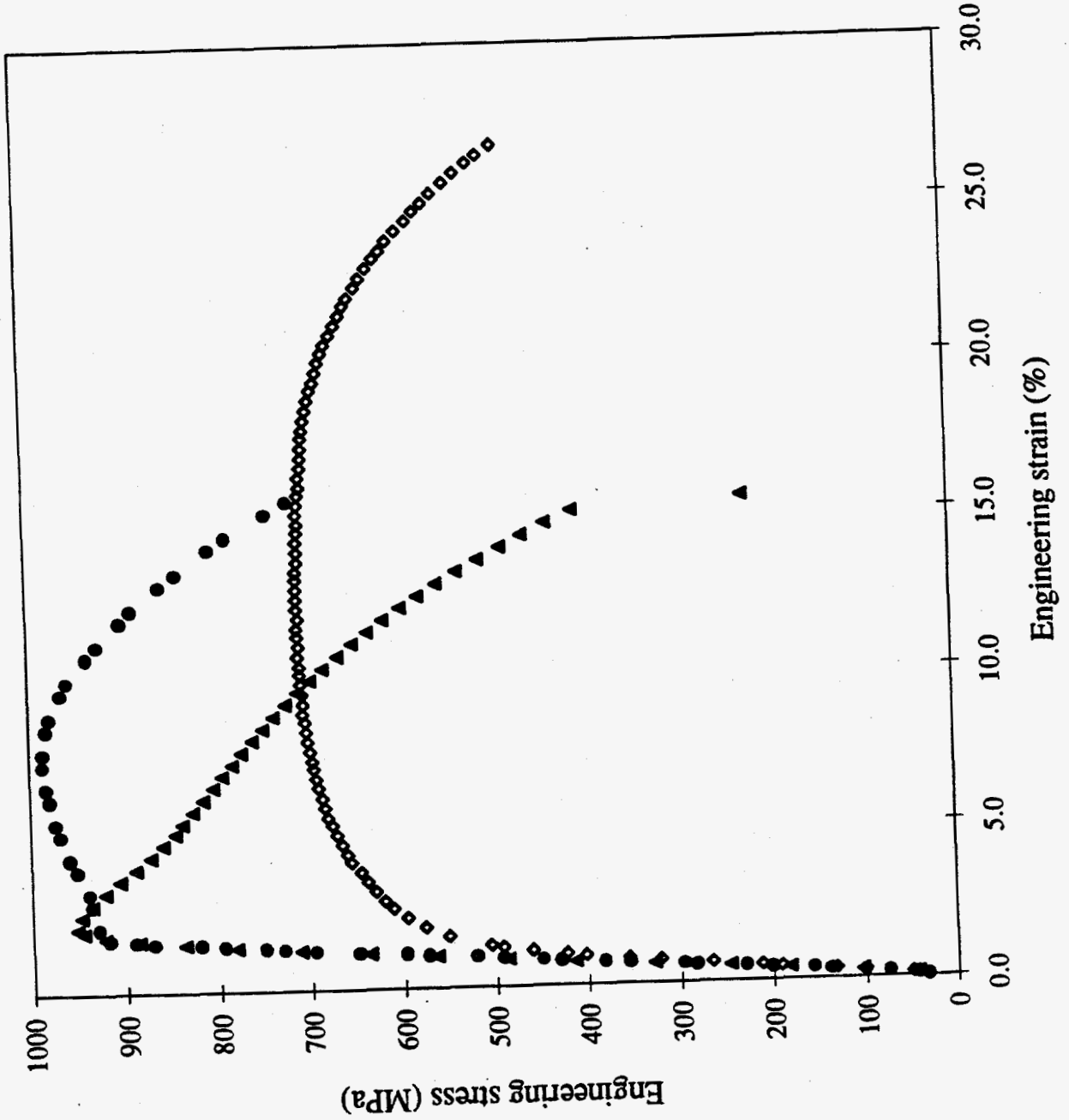
### HT9 ductility



◆ Reference  
■ 1.5 dpa at 350 K (SIWAS-5)  
▲ 2.5 dpa at 573 K (ILAS-2)

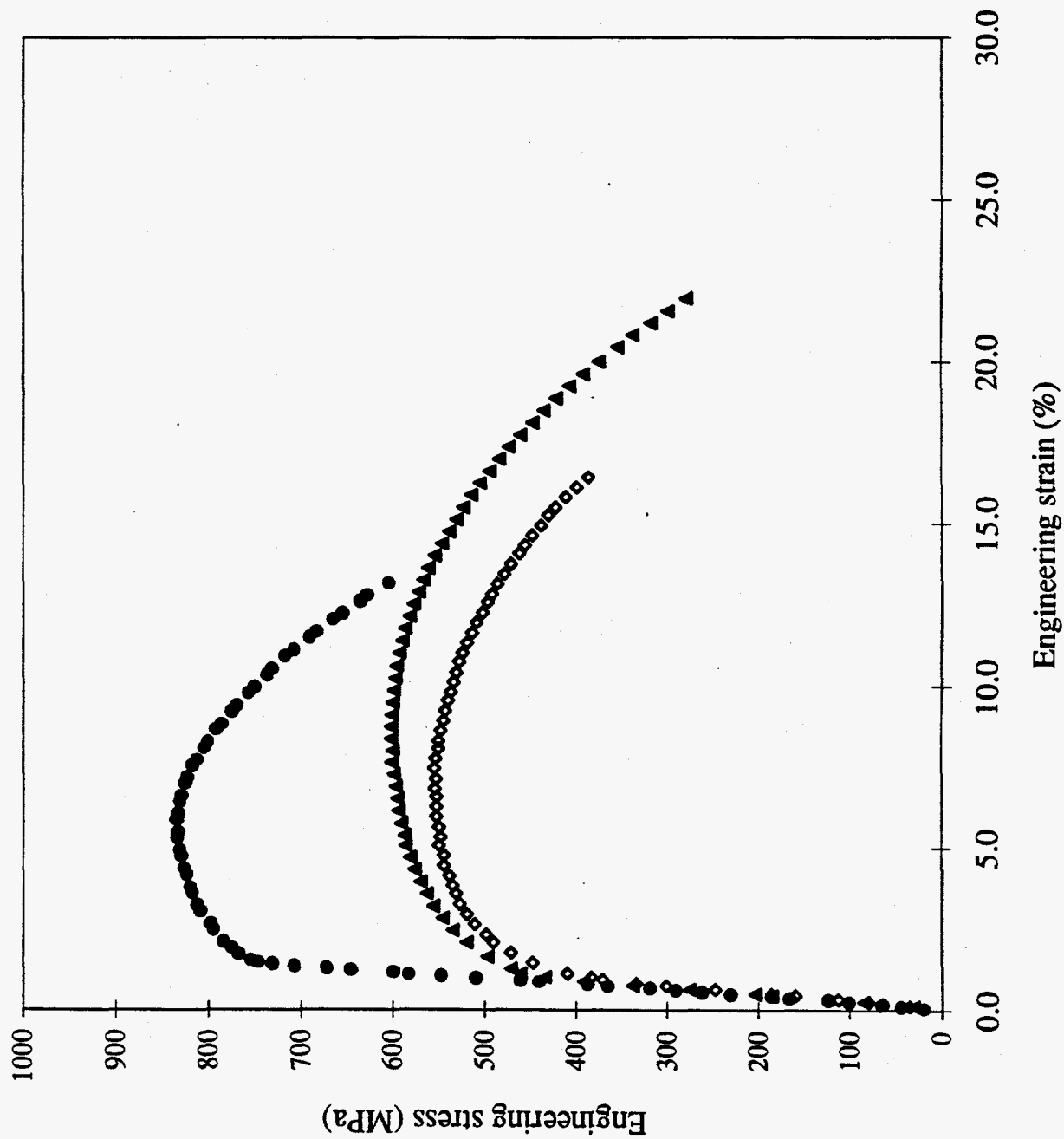
ECN Nuclear Energy

### Tensile curves HT-9 at RT



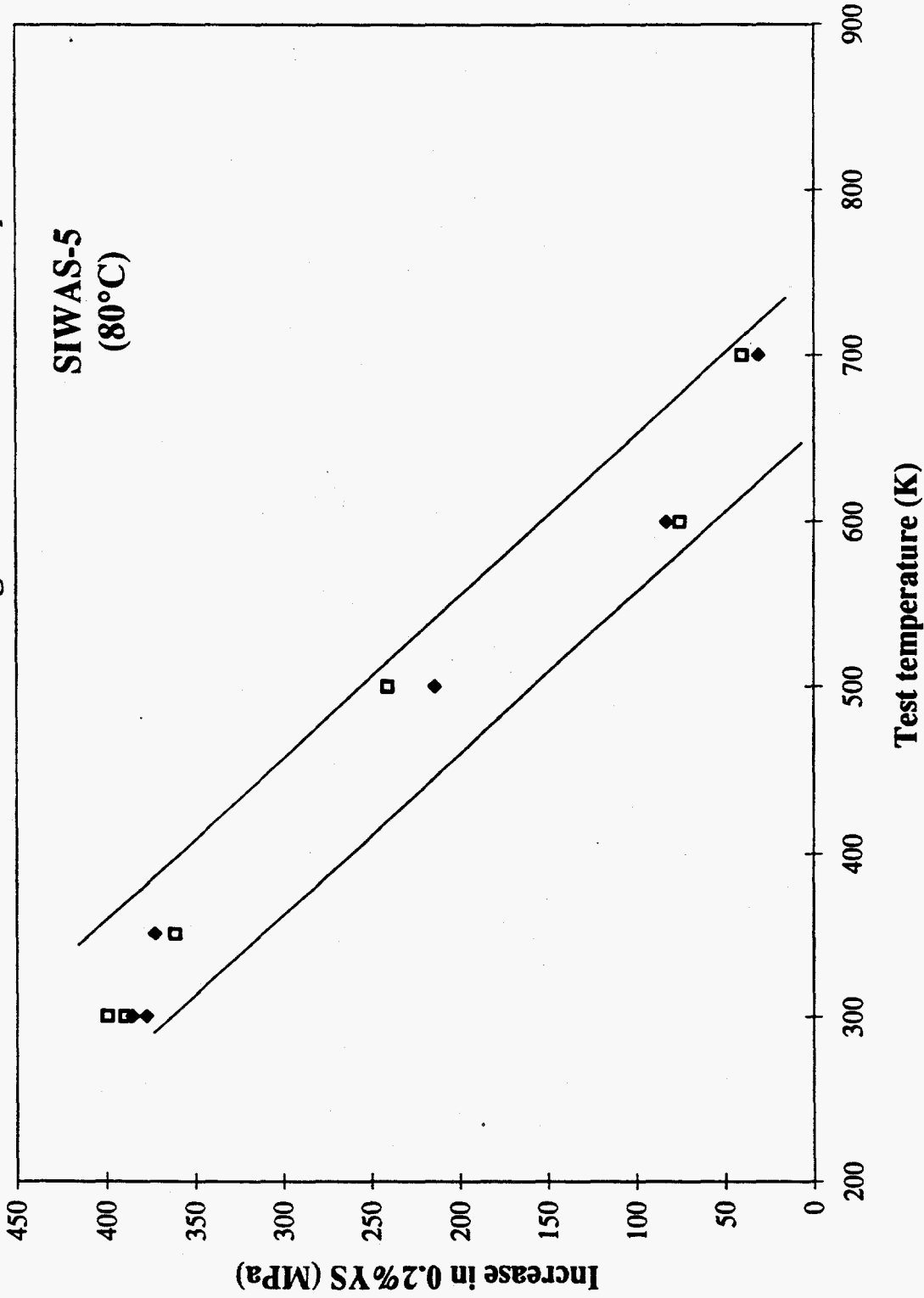
- ◆ Reference
- ▲ 1.5 dpa at 350 K (SIWAS-5)
- 2.5 dpa at 573 K (ILAS-2)

### Tensile curves HT-9 at 673-723 K



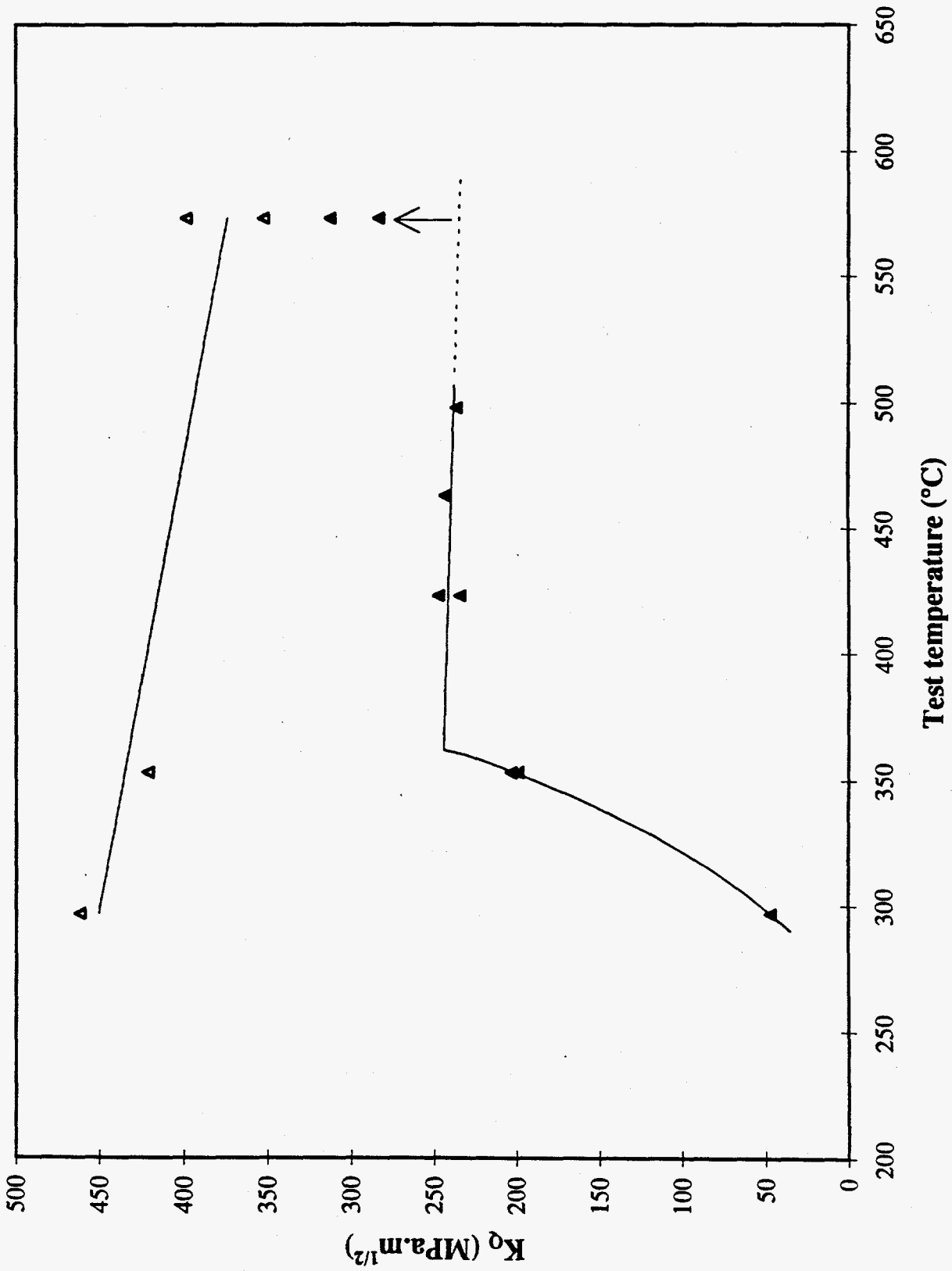
◆ Reference  
▲ 1.5 dpa at 350 K (SIWAS-5)  
● 2.5 dpa at 573 K (ILAS-2)

### Irradiation hardening as function of test temperature



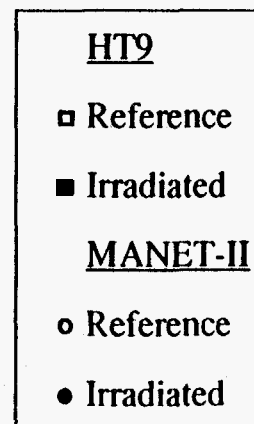
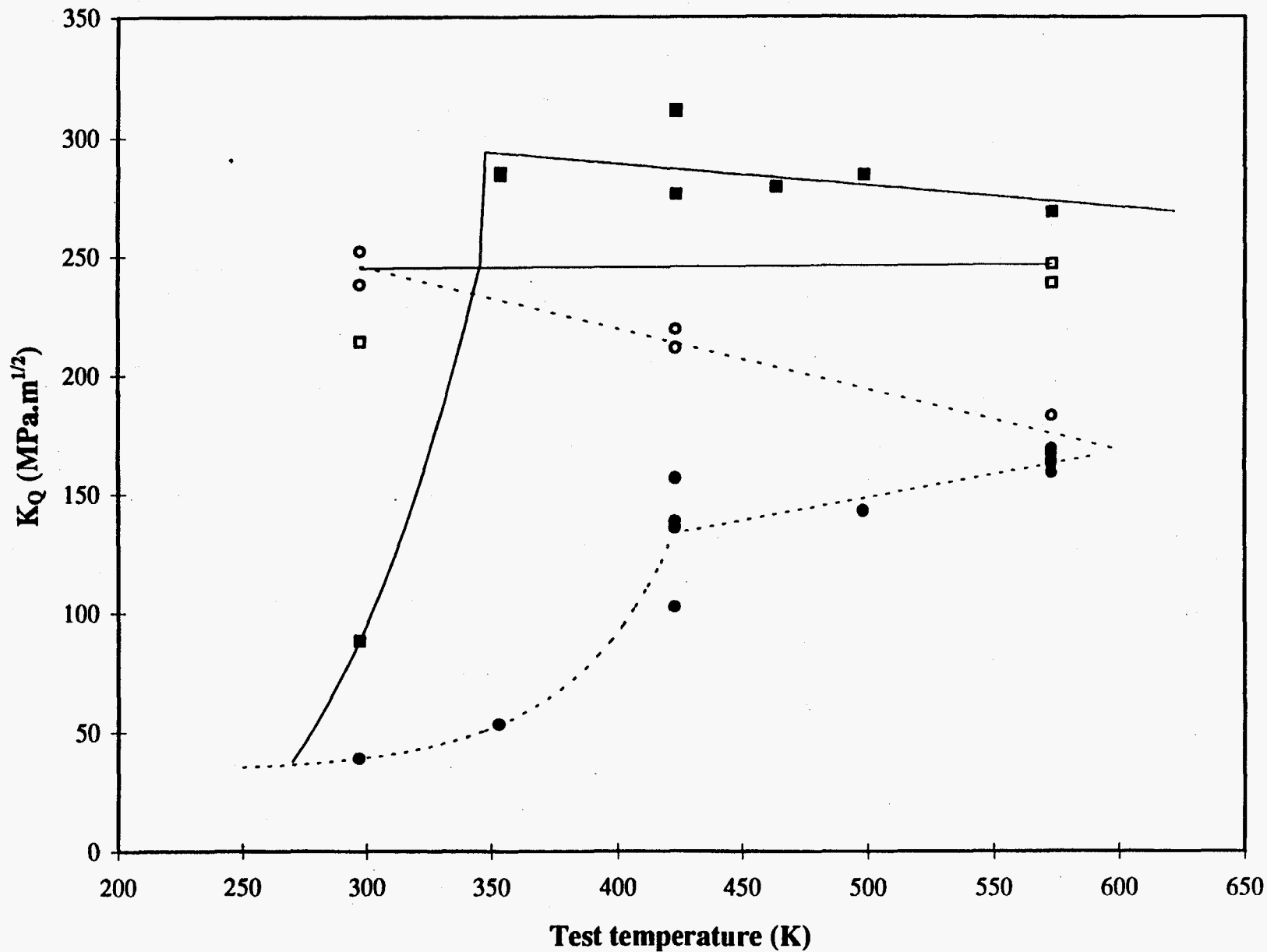
◆ HT-9  
□ Mod. 9%Cr-F

# Mod. 9%Cr steel irradiated to 1.5 dpa at 350 K

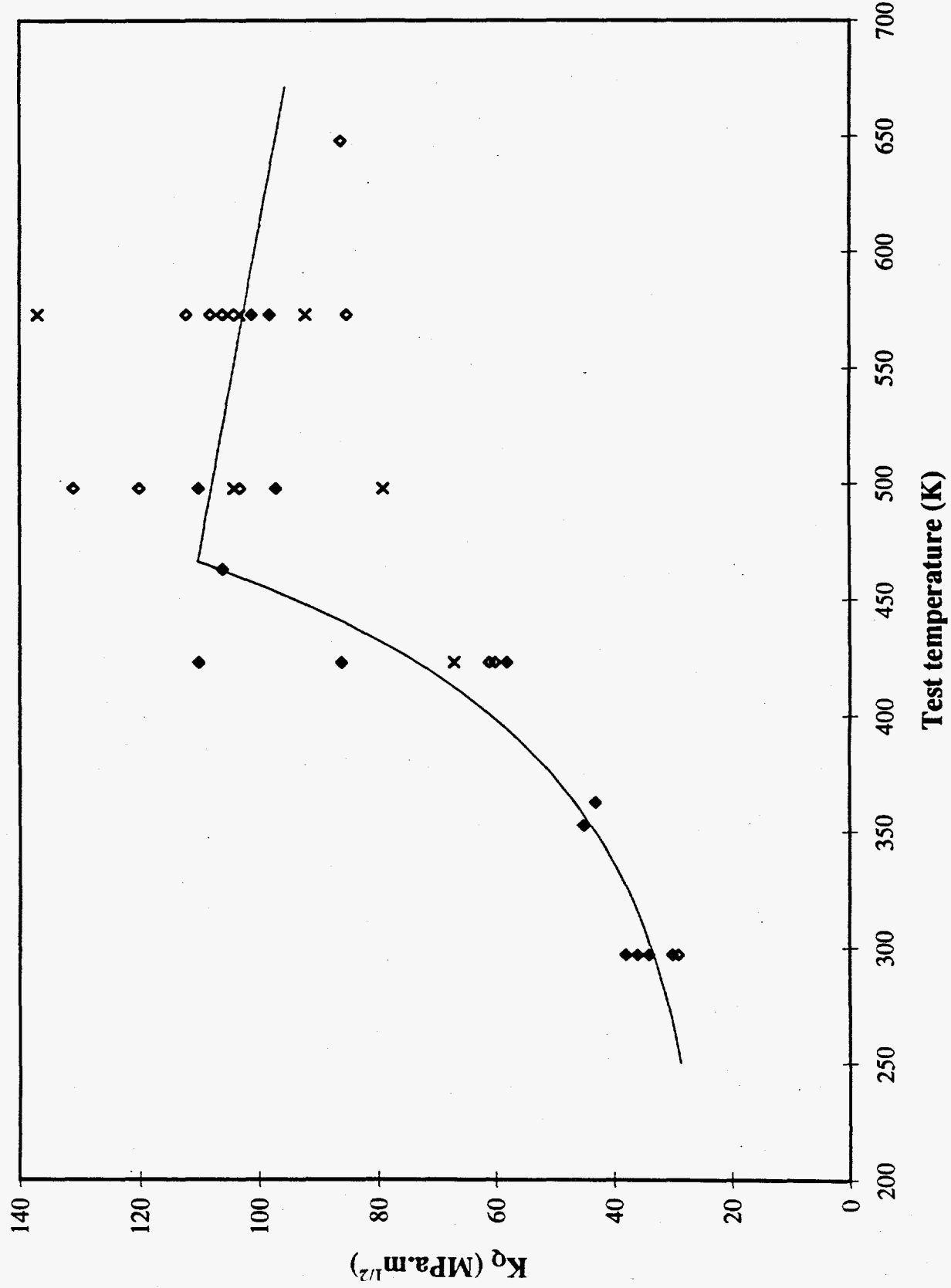




12%Cr steels irradiated to 1.5 dpa at 350 K

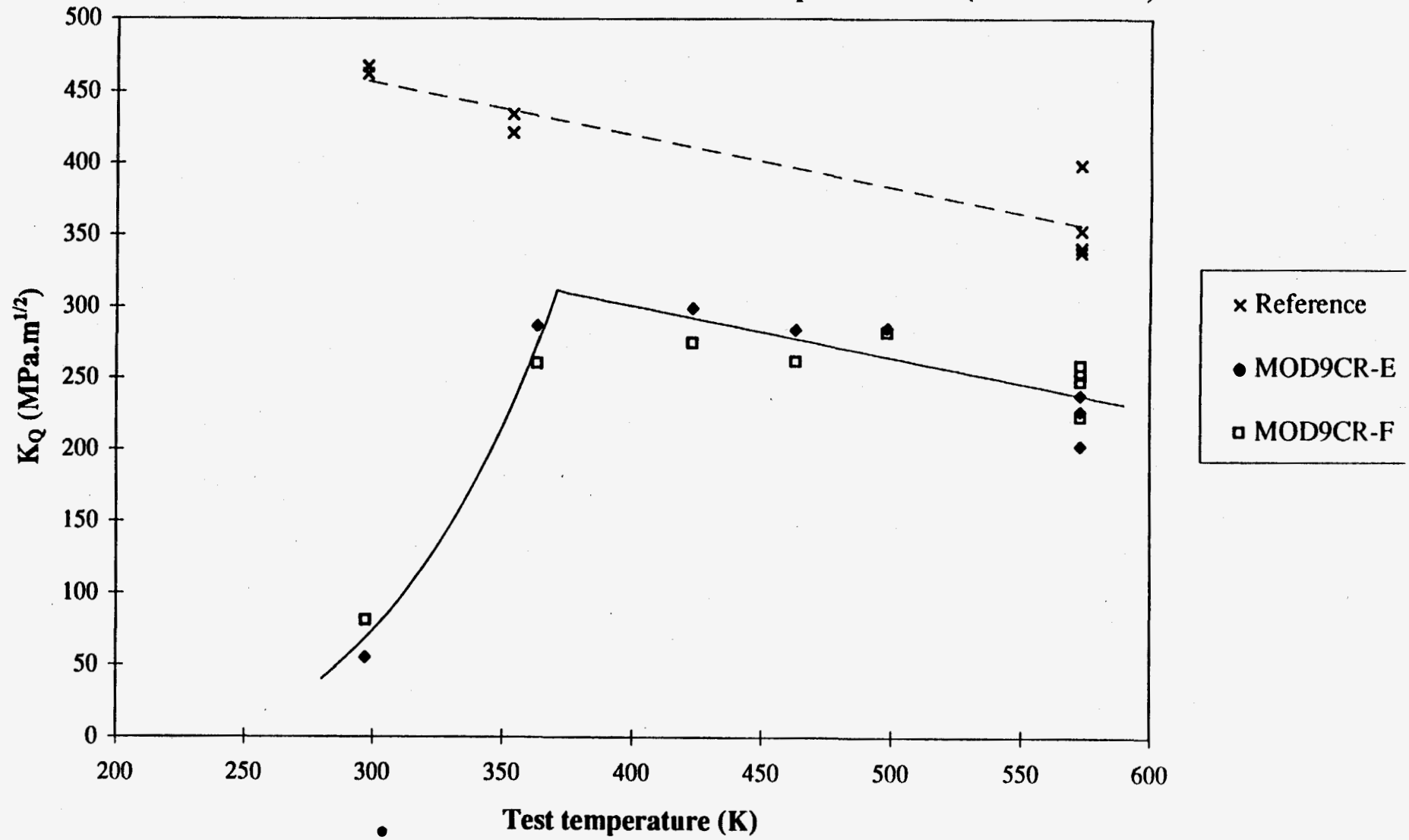


# MANET-II irradiated to 1.5 dpa at 573 K (CATETO-I)

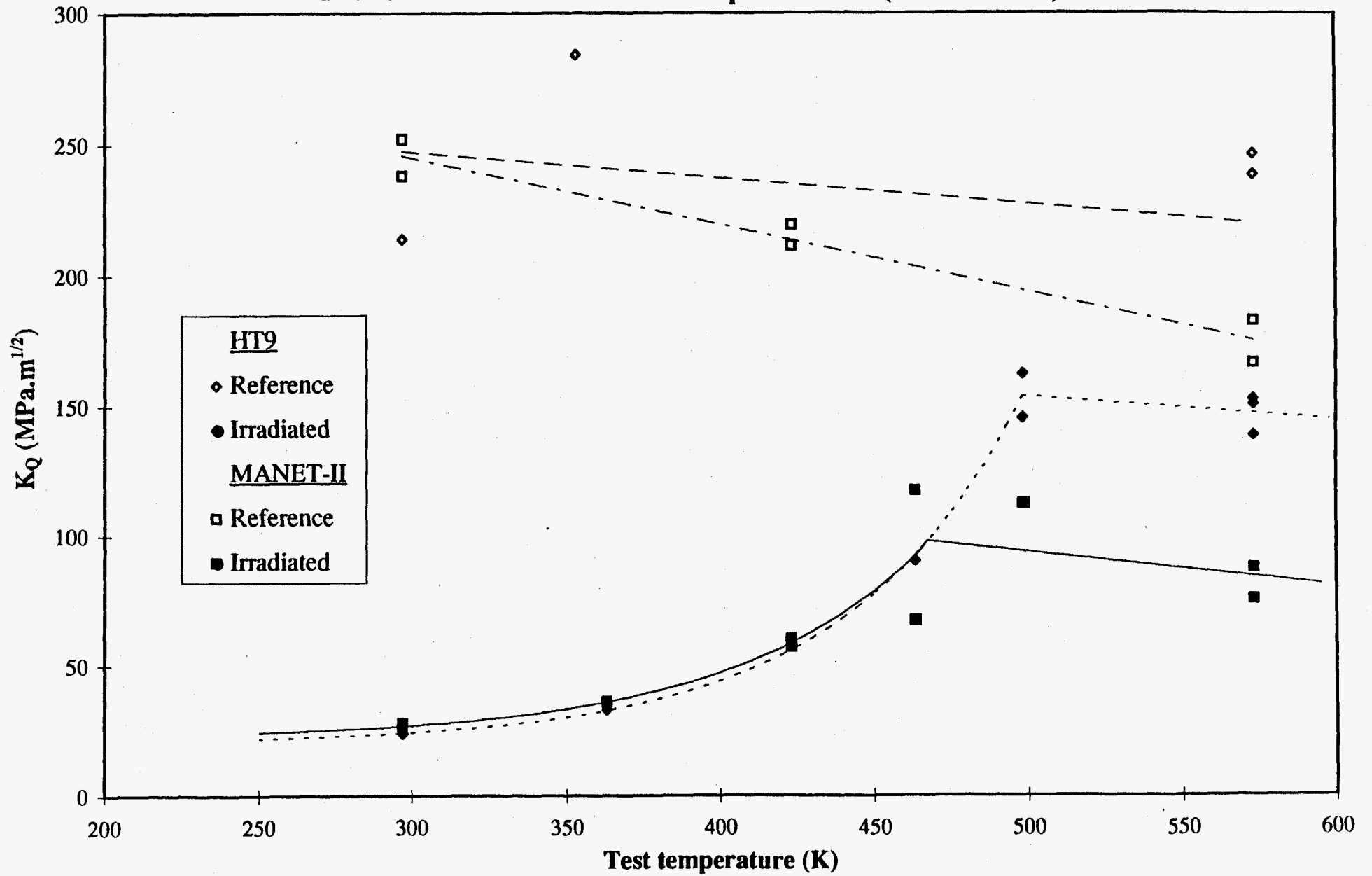


◆  $a/W = 0.55-0.6$   
×  $a/W = 0.6-0.65$   
◊ Side groove

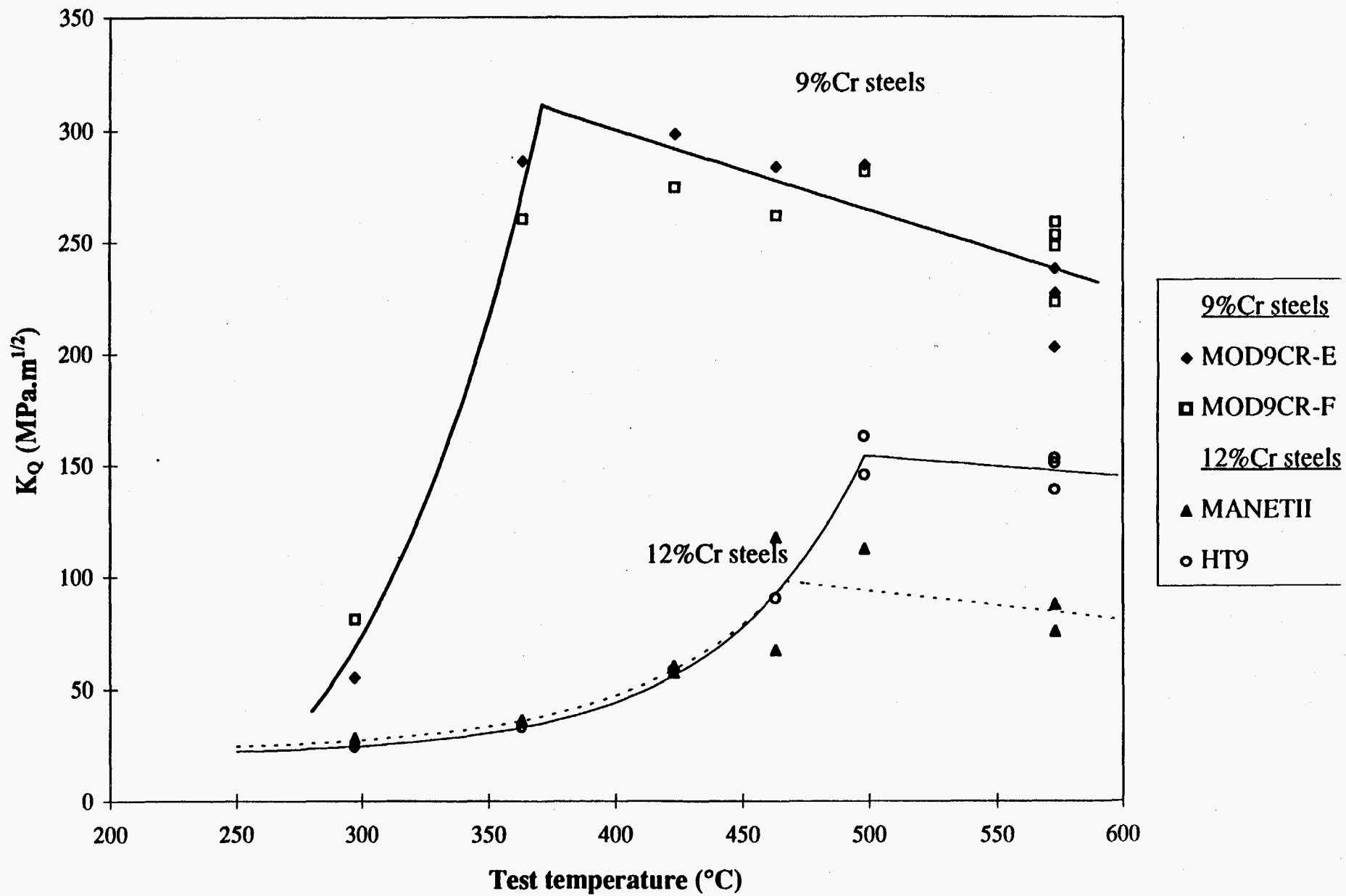
Mod.9%Cr steels irradiated to 2.5 dpa at 573 K (CATETO-II)



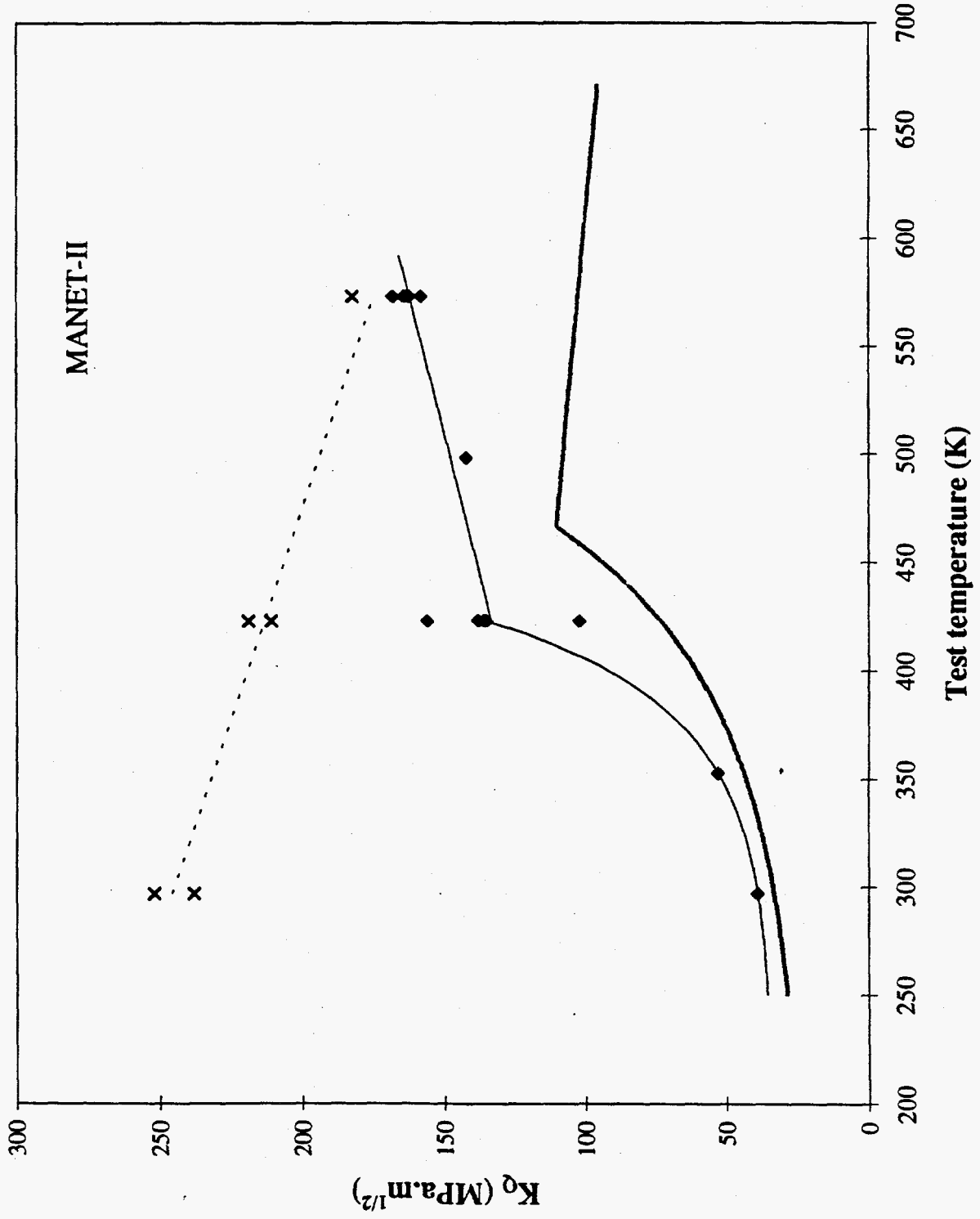
### 12%Cr steels irradiated to 2.5 dpa at 573 K (CATETO-II)



FM steels irradiated to 2.5 dpa at 573 K

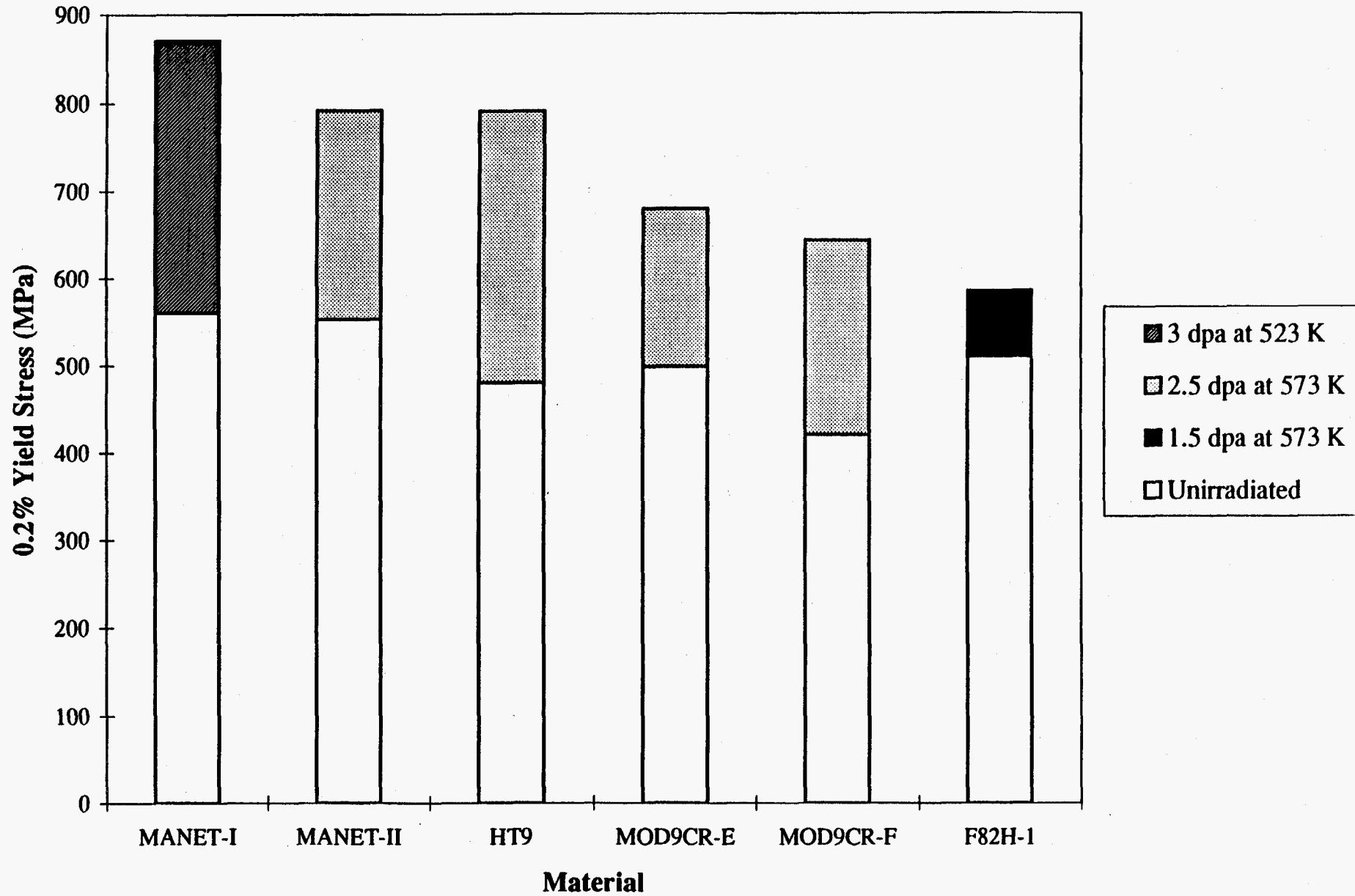


# Effect of irradiation temperature on FT behaviour

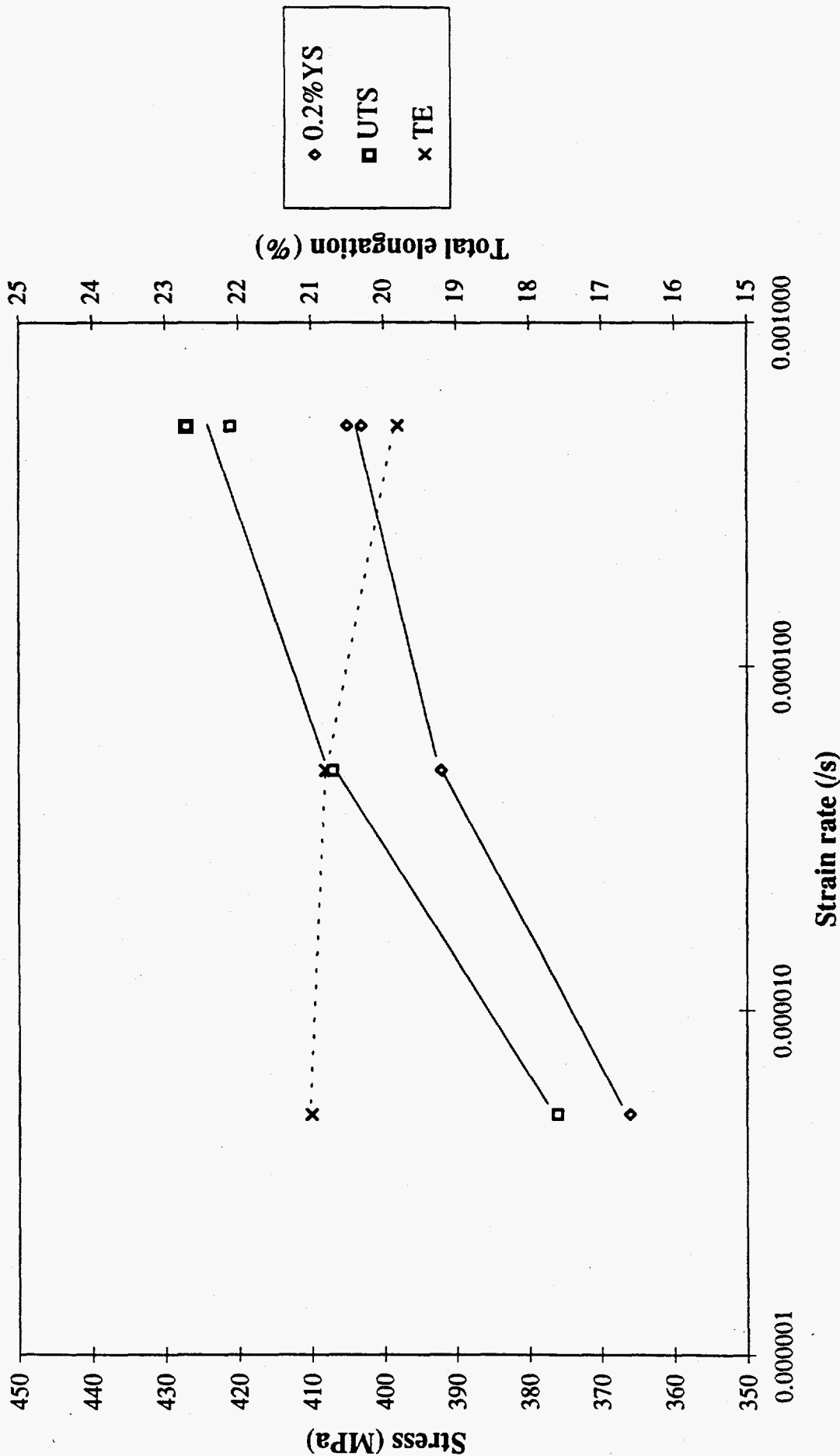


x Reference  
 ♦ 1.5 dpa at 350 K  
 — 1.5 dpa at 573 K

### Irradiation hardening of FM steels

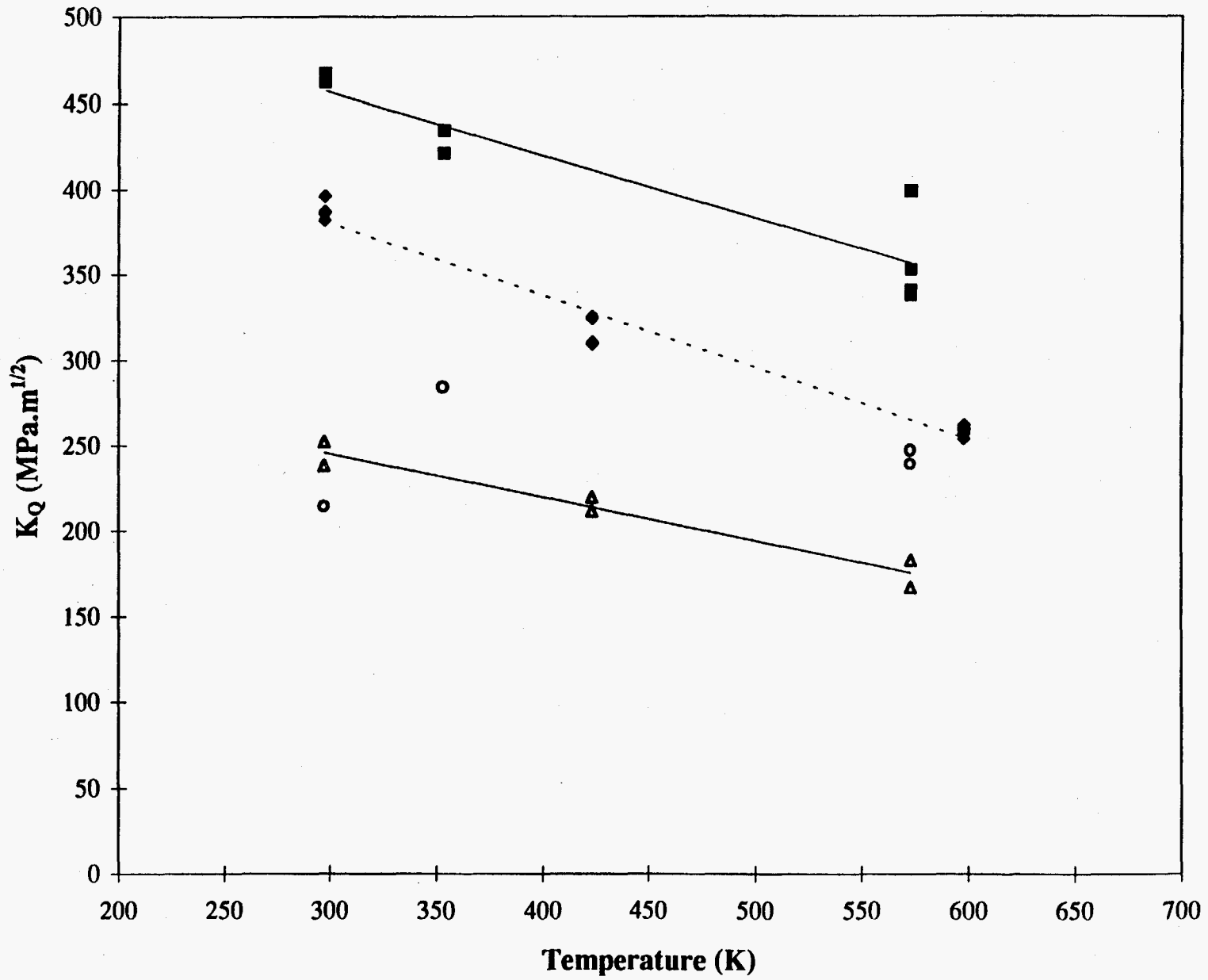


### Tensile test results F82H-mod at 775 K





### Reference FTT curves FM steels



- 9%Cr steels
  - ◆ F82H-mod.
  - Mod.9%Cr
- 12%Cr steels
  - HT9
  - △ MANET-II

## OVERVIEW OF FM IRRADIATION PROGRAMME

ID	Task Name	Material(s)	Specimen	Temperature (K)	Dose (dpa)
1	SIWAS-06	F82H-7.5/15mm, BS	Tensile, CT, KLST	350	2.0
2	SIWAS-07	Mod.9%Cr, BS, F82H-7.5/15mm	Tensile, CT, KLST	350	2.0
3	ILAS-04	F82H-7.5/15mm, JLF-1(B), US-9Cr	Tensile	573	2.5
4	ILAS-05	BS, LA12TaLC	Tensile	573	2.5
5	CHARIOT-02	F82H-7.5/15mm	CT, KLST, disks, mini-tens.	573	2.5
6	CHARIOT-03	BS, Mod.9%Cr, LA12TaLC	CT, KLST	573	2.5
7	ILAS-06	EB & TIG F82H-15/25mm	Tensile, lcf	573	2.5
8	ILAS-07	EB & TIG F82H-15/25mm, Mod.9%Cr, BS	Tensile, lcf	573	10
9	CHARIOT-04	F82H-25mm, incl. opt. heat treat.	CT, KLST, coupons	573	2.5
10	CHARIOT-05	EB & TIG of F82H-15/25mm	CT, KLST	573	2.5
11	CHARIOT-06	F82H-7.5/15/25mm, BS	CT, KLST, disks, mini-tens	573	10
12	CHARIOT-07	EB & TIG of F82H-15/25mm	CT, KLST	573	10

### OVERVIEW OF FM IRRADIATION PROGRAMME

ID	Task Name	Temperature (K)	Dose (dpa)	1995	1996	1997	1998	1999	2000	2001
1	SIWAS-06	350	2.0		■					
2	SIWAS-07	350	2.0			■				
3	ILAS-04	573	2.5	■						
4	ILAS-05	573	2.5		■					
5	CHARIOT-02	573	2.5		■					
6	CHARIOT-03	573	2.5			■				
7	ILAS-06	573	2.5				■			
8	ILAS-07	573	10				■			
9	CHARIOT-04	573	2.5				■			
10	CHARIOT-05	573	2.5				■			
11	CHARIOT-06	573	10				■			
12	CHARIOT-07	573	10				■			

# Joint work

---

- ◆ Microstructure: PNL (US) TEM  
UCSB (US) SANS
- ◆ Small specimen
  - tensile: PNL (US)
  - FT (PCKLST): UCSB (US)
- ◆ Welds: CEA Saclay (F)
- HIP material: CEA Grenoble (F)

# Conclusions

---

- ◆ High temperature irradiation damage more stable than at low temperature, valid for both 9%Cr and 12%Cr steels
- ◆ Toughness of Mod.9%Cr steels is favourable over 12%Cr steels, both in unirradiated and irradiated condition
- ◆ High dose irradiation behaviour at temperatures below 573 K unknown, above 600 K *in situ* annealing of damage

CRPP Data on the Program of the  
Effects of Radiation Hardening and  
He/dpa effects on F82H

P. Spätig

CRPP - Fusion Technology  
PIREX Materials Group  
EPFL -Switzerland

IEA Working Group on Ferritic Steels

Jet Headquarters, Culham, UK  
24-25 October 1996

## Objectives

- \* Study of the effects of radiation hardening and helium in a low activation steels. In the PIREX facility we have a bulk helium production
- \* Create a large irradiation and testing matrix in which we will attempt to separate the helium effects from those of radiation hardening. The KfK Dual Beam, the ~~Studvik~~ reactor and PIREX facilities are involved. *Studvik*
- \* Effects of radiations hardening at different doses and four temperatures 40°C, 250°C, 350°C and 450°C
- \* Post-irradiation tensile properties

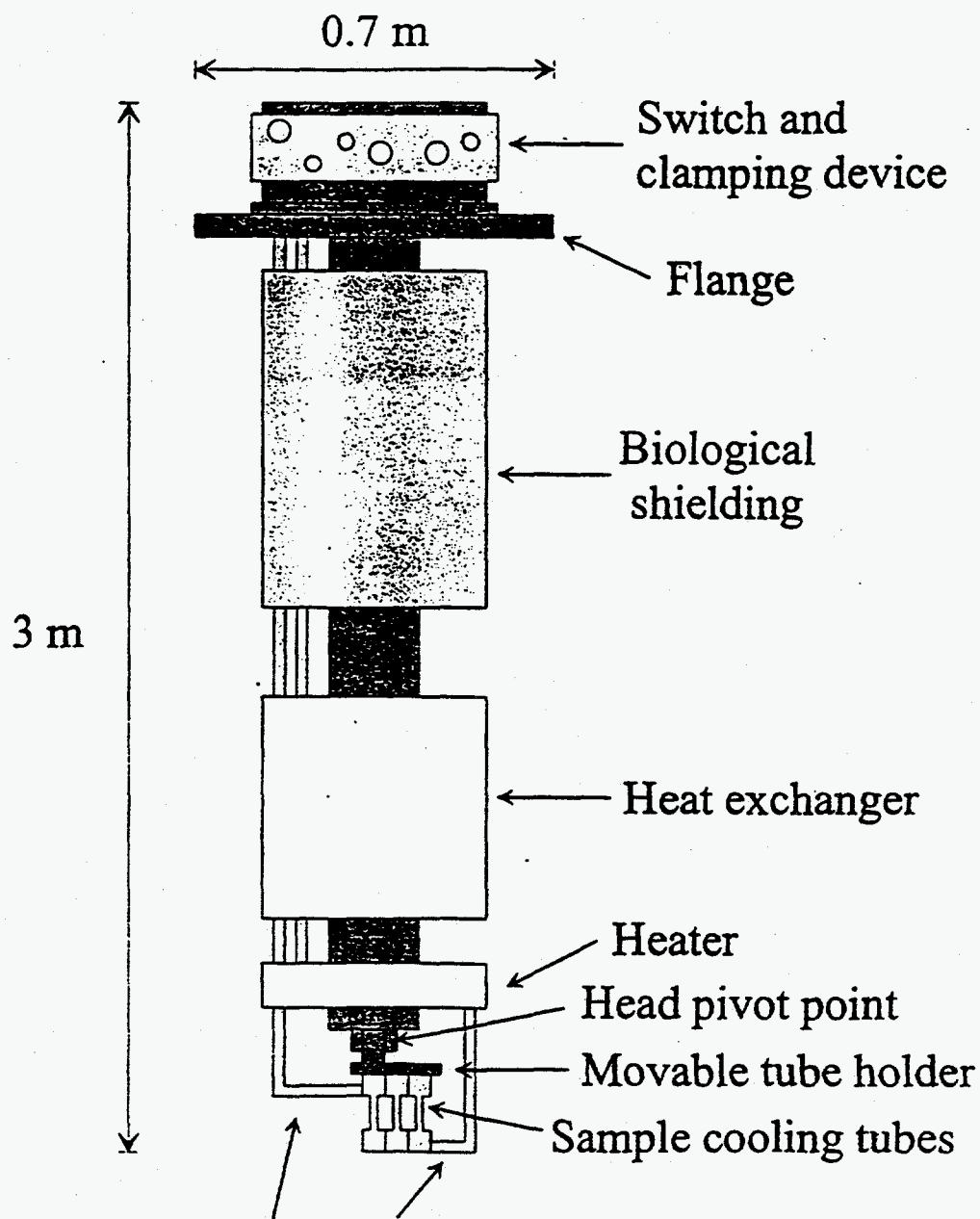
~~Studvik~~ reactor: radiations hardening on pre-irradiated PIREX specimens and virgin specimens (*Studvik - Sweden*)

PIREX: The 590 MeV creates dpa and He appm in such a way that for a dose of about 1 dpa corresponds a creation of 100 appm He in ferritic/martensitic steels

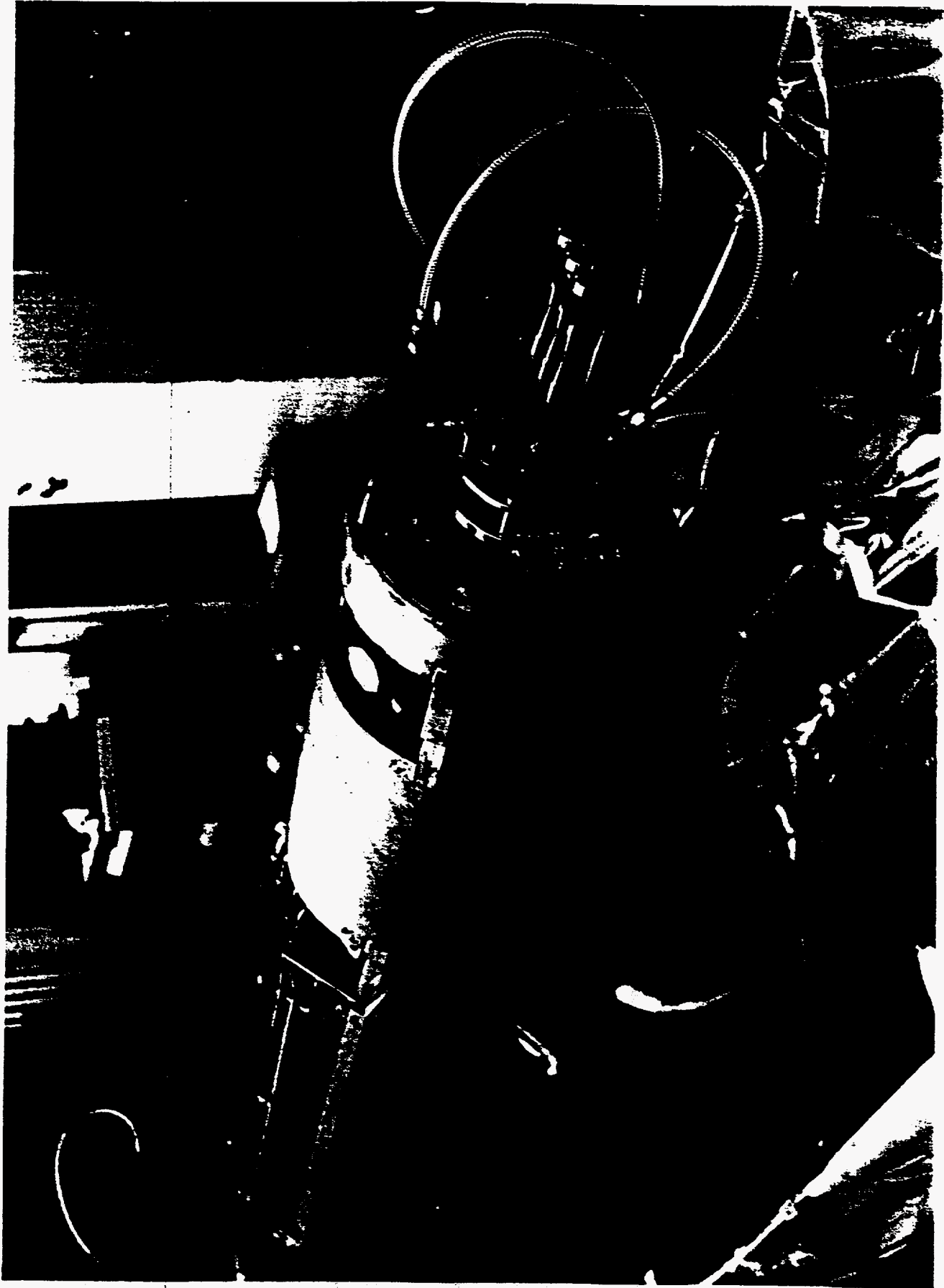
KfK dual beam: dpa and controlled amount of He

## PIREX facility

- \* Beam line optics provides beams in the range  $4\sigma_x = 1$  to 3 mm and  $4\sigma_y = 3$  to 10 mm. Uniformity is improved by the use of a beam wobbler.
- \* Extraction of the deposited heat in the target (3 to 11 kW/cm<sup>3</sup>) with helium gas cooling.







## Proton interactions

Medium energy protons interact directly with the nucleons within the nucleus rather than with the nucleus as a whole.

The interaction is described by the **intranuclear cascade - evaporation model**:

**Intranuclear cascade:** the struck protons or neutrons in the nucleus have sufficient energy to escape from it. The additional energy deposited leaves the nucleus in an excited state.

**Evaporation:** the nucleus left in an excited state, decays to a ground state by the isotropic emission of light particles : protons, deuterons, tritons, helium 3 and 4. These particles have energies of a few MeV.

The **recoiling nucleus** is the main agent of displacement damage.

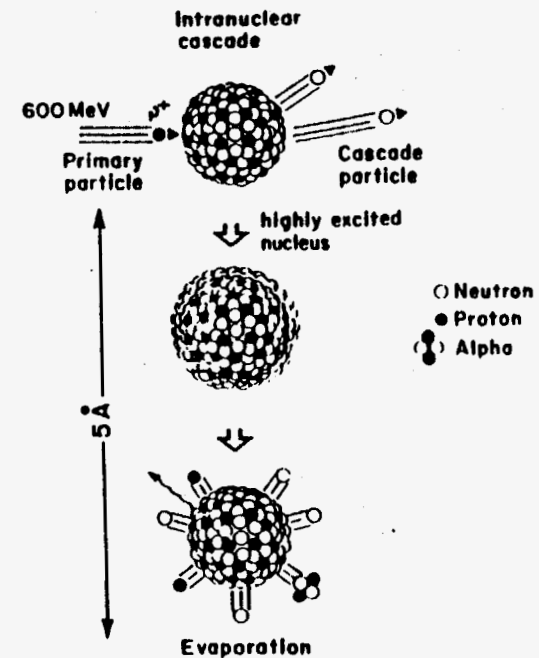
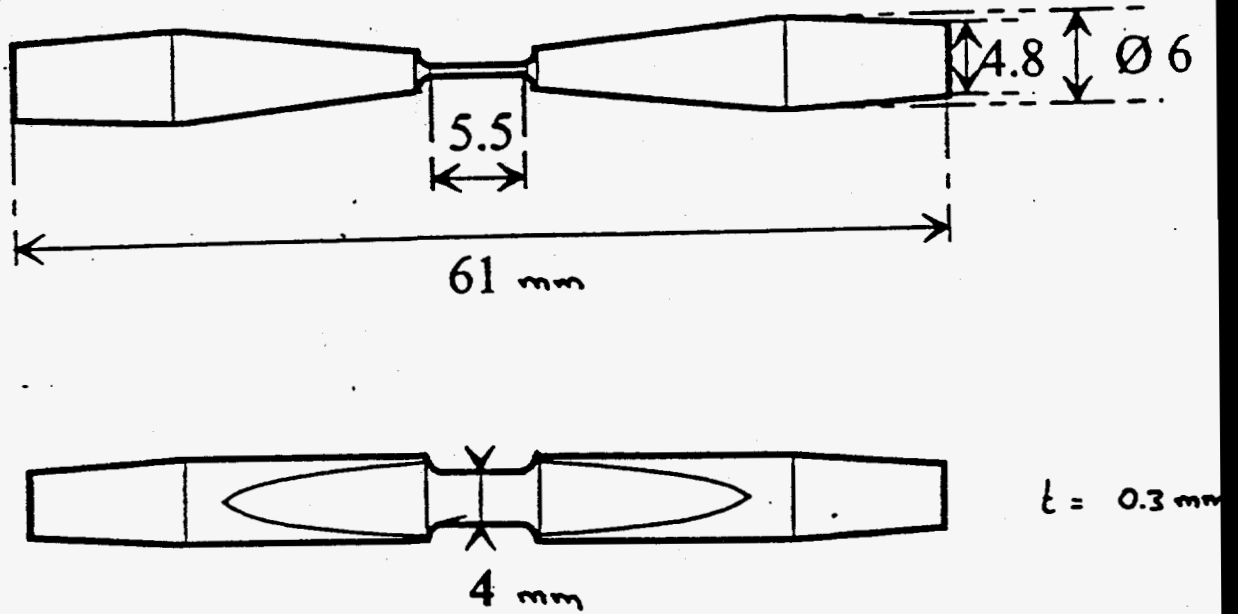
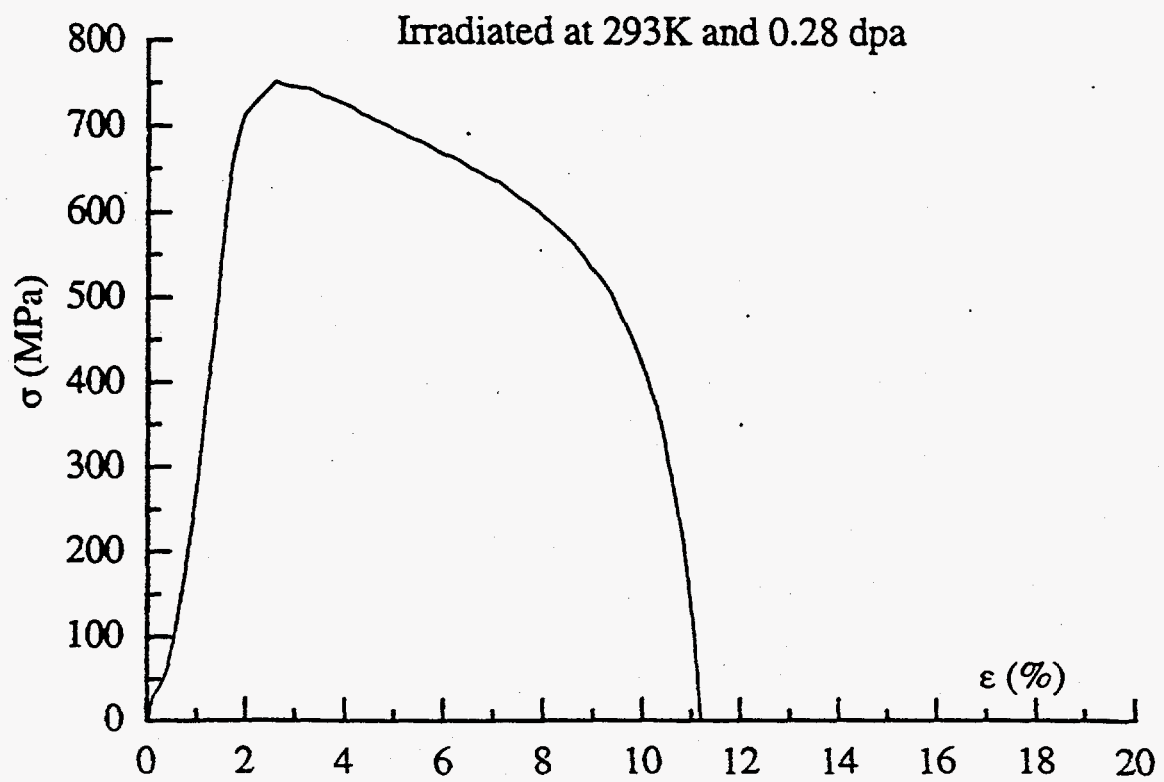
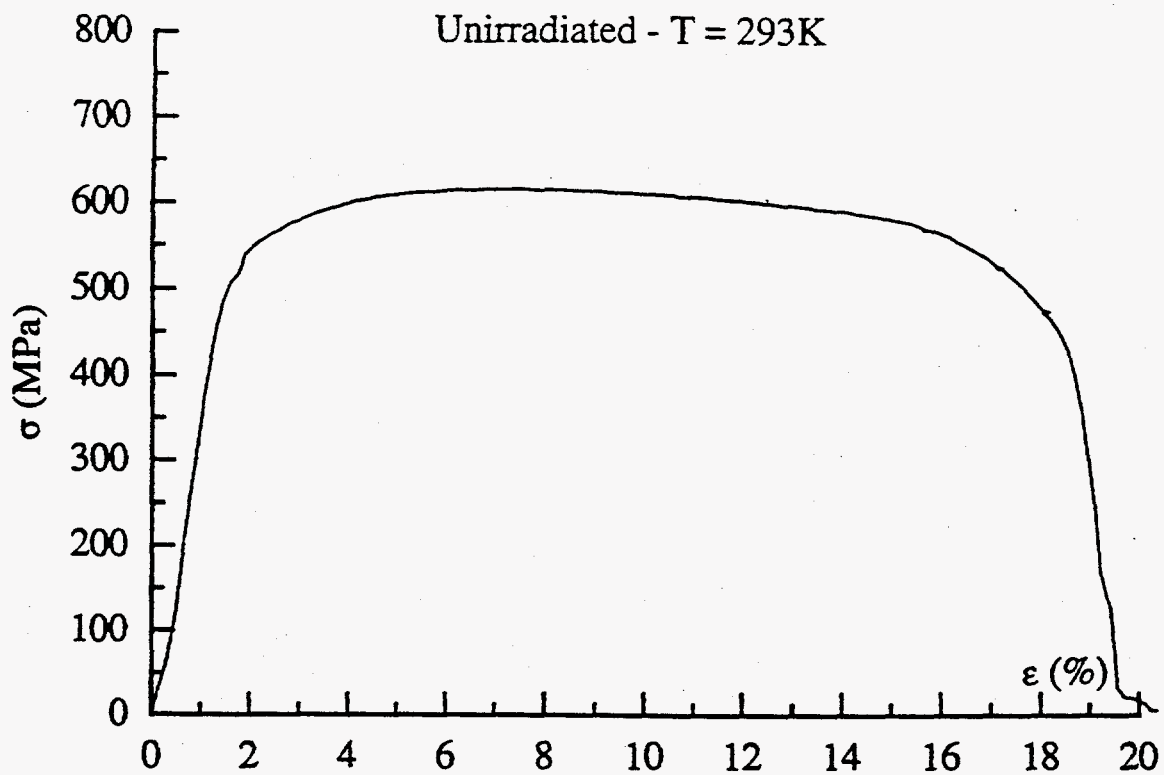


Fig. 1. Intranuclear cascade-evaporation model. The flight path from start to finish is 5 Å or less.

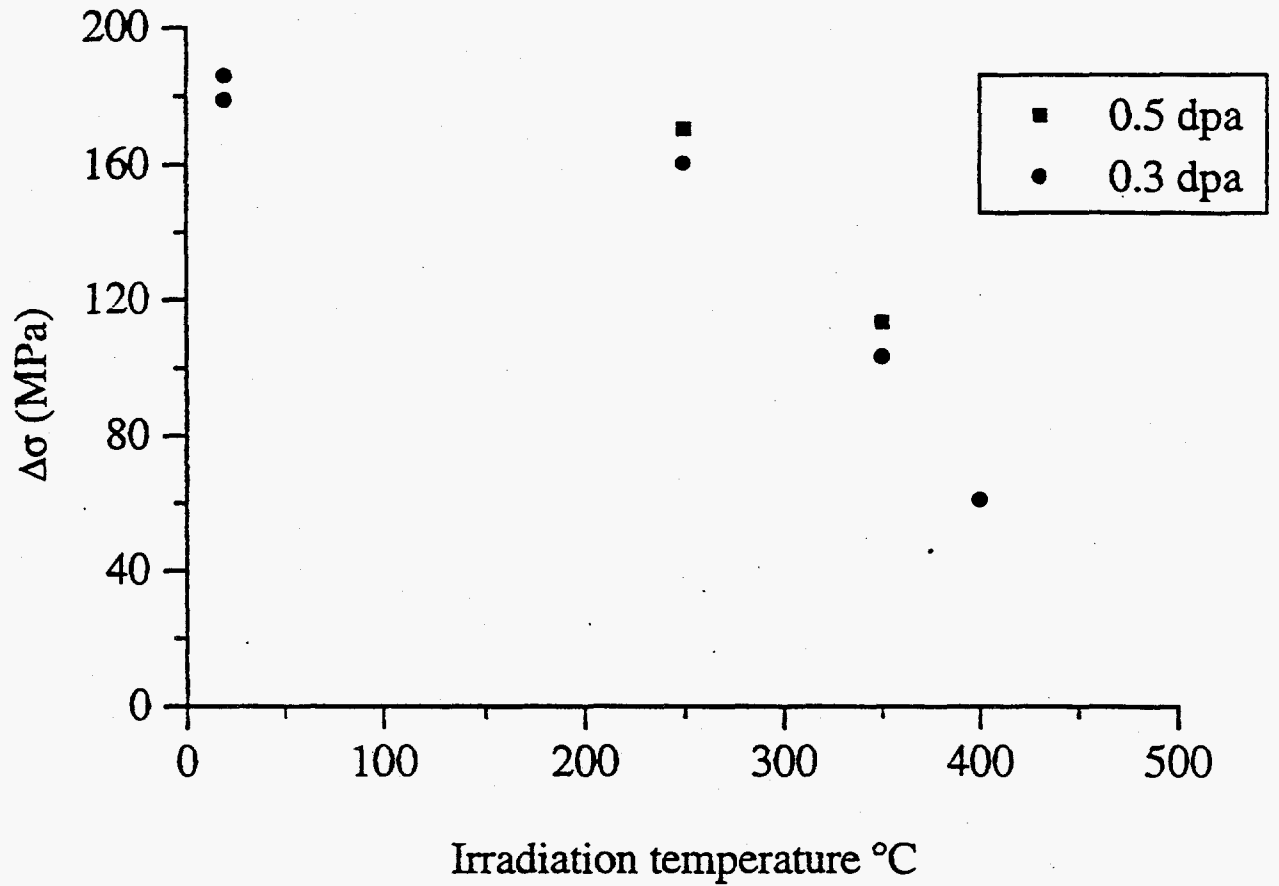
## PIREX tensile specimens



Geometry of the PIREX specimens which are irradiated at the present time  
(e.g. low activation steel F82H)

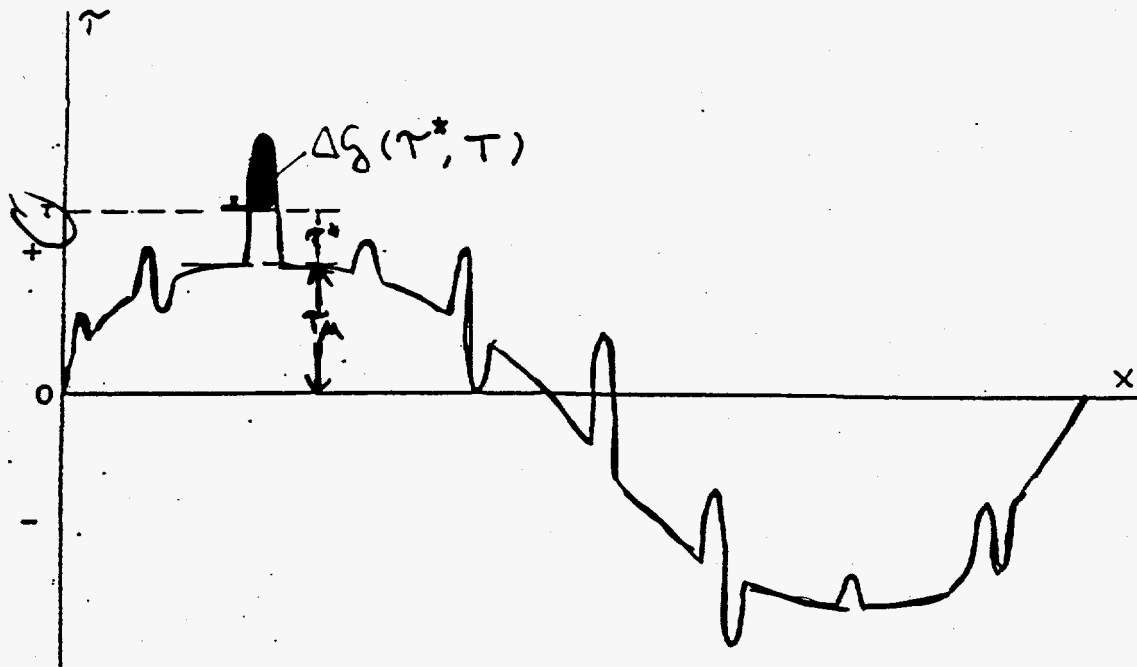


Irradiation hardening  $\Delta\sigma$  (MPa)  
 $\Delta\sigma$  measured at room temperature



\* at these doses (0.5dpa and 0.3 dpa) the production of helium is about 20 and 30 appm. A small effect of helium is expected.

## Force on the moving dislocations



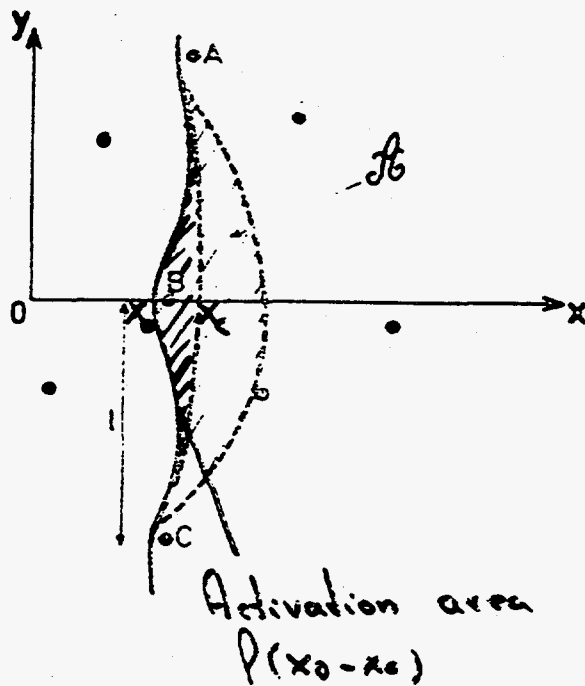
Schematic representation of the variation of the force on a dislocation with distance along the slip plane. The small peaks corresponds to thermal obstacles.

- $F_\mu$  or  $(\tau_\mu)$  long-range force varies slowly with the position of the dislocation on the slip plane. Due to large obstacle such as second phase particles, dislocations on parallel-slip planes, etc...
- $F^*$  or  $(\tau^*)$  short-range force which acts over a few atomic diameters only due for instances to the forest dislocations, point defects, tetragonal defects, etc

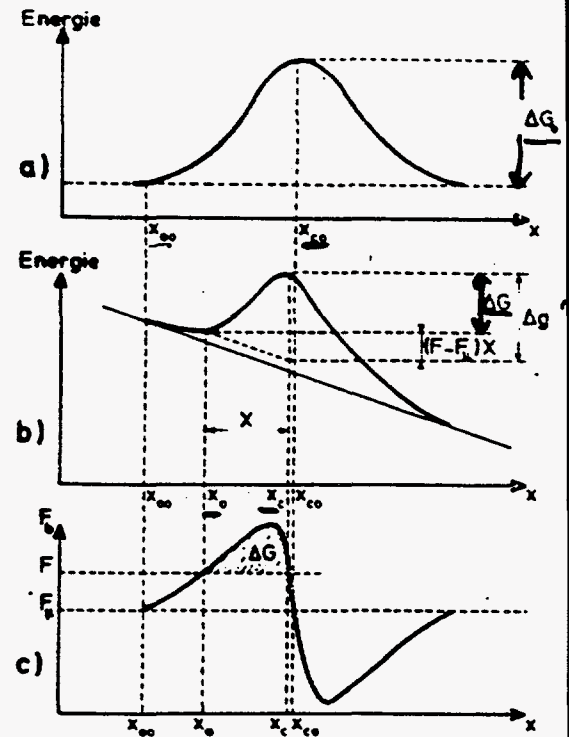
## Decomposition of the applied stress $\tau_a$ :

$$\tau_a = \tau^*(\dot{\gamma}_p, T) + \tau_\mu(\gamma_p)$$

# Thermal activation - Energy of activation



Interaction between the dislocation and a localised obstacle



Energy and force as a function of the distance

The force of the obstacle is defined by:  $\tau_b = \frac{1}{b} \left( \frac{\partial \Delta G}{\partial x} \right)_{\tau = \tau_m}$

and the Gibbs free energy is:  $\Delta G = \int_{x_0}^{x_c} (\tau_b(\tau, x) + \tau_m - \tau) b dx$

Let us define:  $\Delta g(\tau, \tau^*) = \int_{x_c}^x \tau_b(\tau, x) b dx$

The  $\tau^*$  dependence comes from  $X(\tau^*, T) = x_c - x_0$

## Strain-rate sensitivity of the flow stress

Thermally activated plastic flow:  $\dot{\gamma}_p = \alpha \rho_m b v = \dot{\gamma}_0 \exp\left(-\frac{\Delta G(\tau^*, T)}{kT}\right)$

Activation volume:  $V_{\text{eff}} = -\frac{\partial \Delta G}{\partial \tau^*}$

$$= (x_0 - x_c) \rho b = \text{Activation area} \times b$$

$$V_{\text{exp}} = kT \frac{\partial \ln \dot{\gamma}}{\partial \tau} = kT \left[ \frac{\partial \ln \dot{\gamma}_0}{\partial \tau} - \frac{\partial \Delta G}{\partial \tau^*} \frac{\partial \tau^*}{\partial \tau} \frac{1}{kT} \right]$$

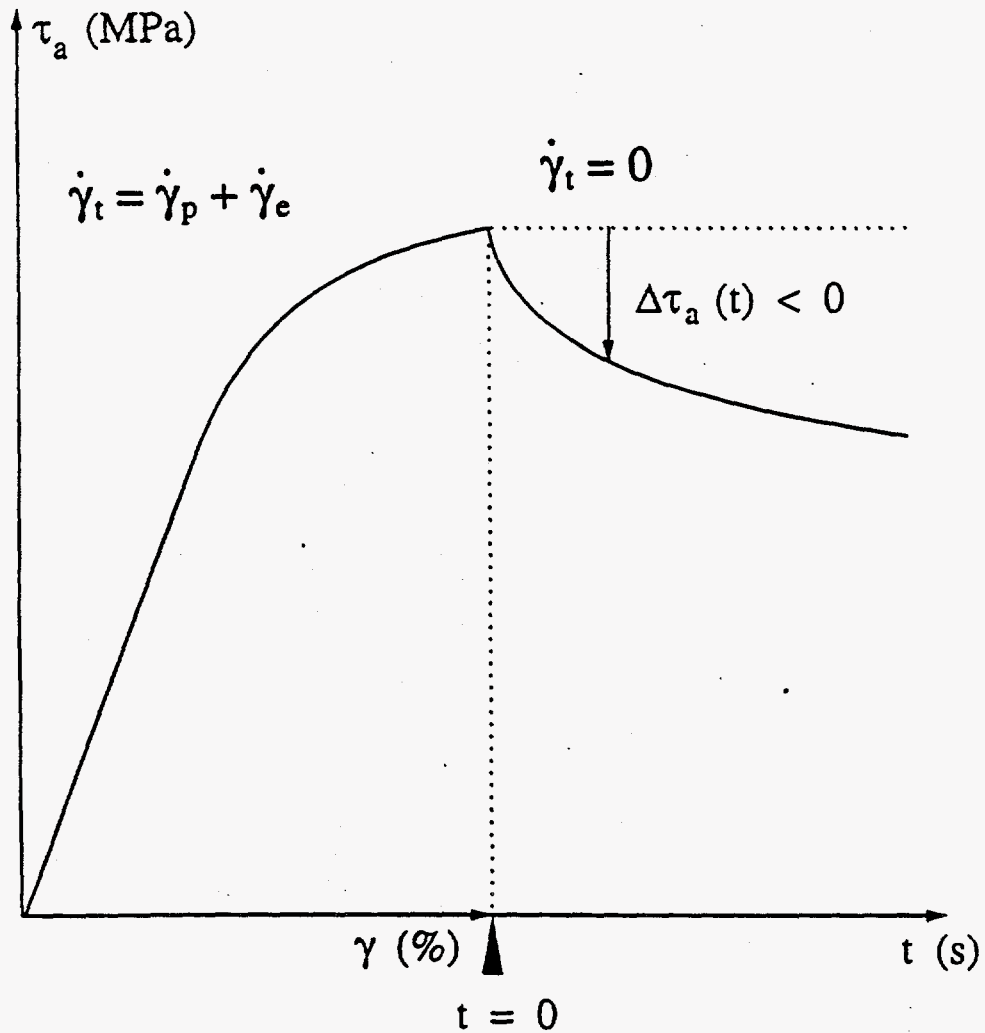
$$V_{\text{exp}} = \underbrace{kT \frac{\partial \ln \dot{\gamma}_0}{\partial \tau}}_{\beta} + \underbrace{V_{\text{eff}} \frac{\partial \tau^*}{\partial \tau}}_{K+1}, \quad V_{\text{eff}} := -\frac{\partial \Delta G}{\partial \tau^*}$$

$V_{\text{eff}}$  is characteristic of a given rate controlling process as well as its stress dependence.

$V_{\text{eff}}$  essentially represents the number of atoms implied during the thermally activated events.



Simple relaxation test:

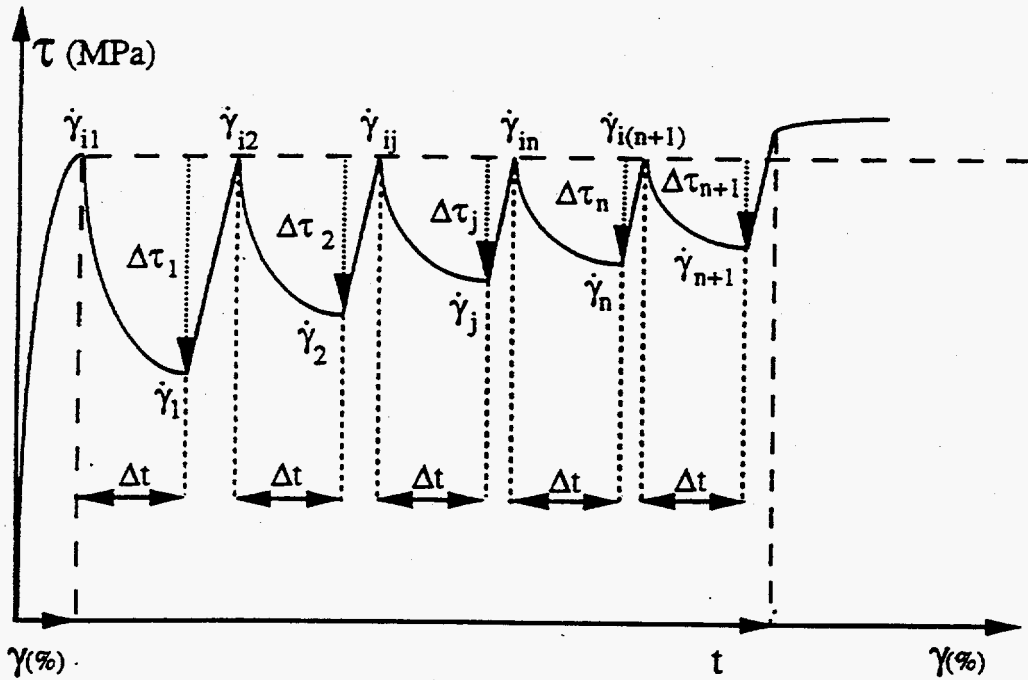


During the test:  $\dot{\gamma}_t = 0$  or:

$$\dot{\gamma}_p = -\dot{\gamma}_e = -\frac{\dot{\tau}_a}{M} = \alpha\rho_m(\Delta\tau^*)bv(\Delta\tau^*)$$

$M$  is the elastic modulus of the specimen-machine assembly

## Activation volume measurement by repeated stress relaxation (Spätig *et al.* 1993)



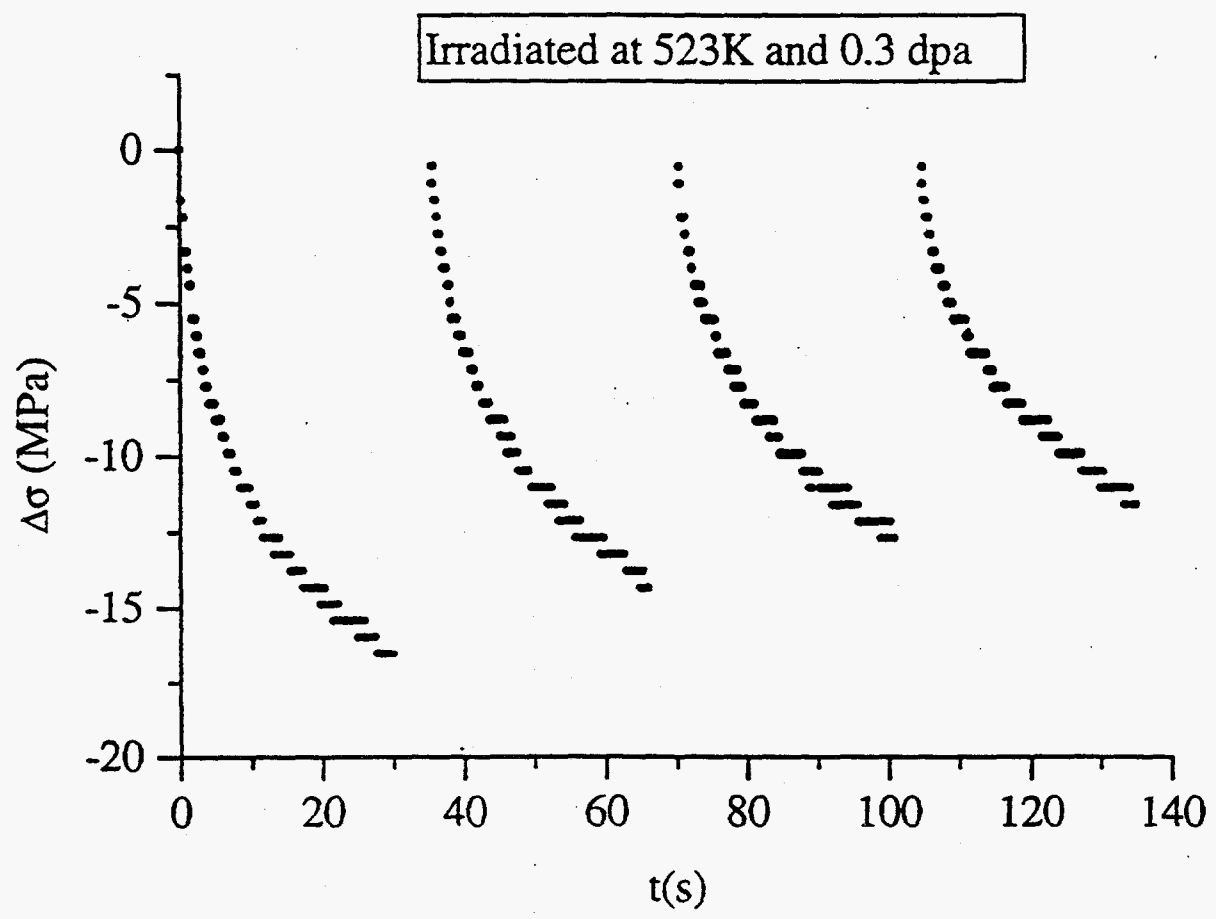
First relaxation yields:

$$\Delta\tau_a(t) = -\frac{kT}{V_a} \ln\left(1 + \frac{1}{c}\right) \quad \text{ou} \quad V_a = V_{\text{eff}}\left(1 + \frac{K}{M}\right)(1 + \beta)$$

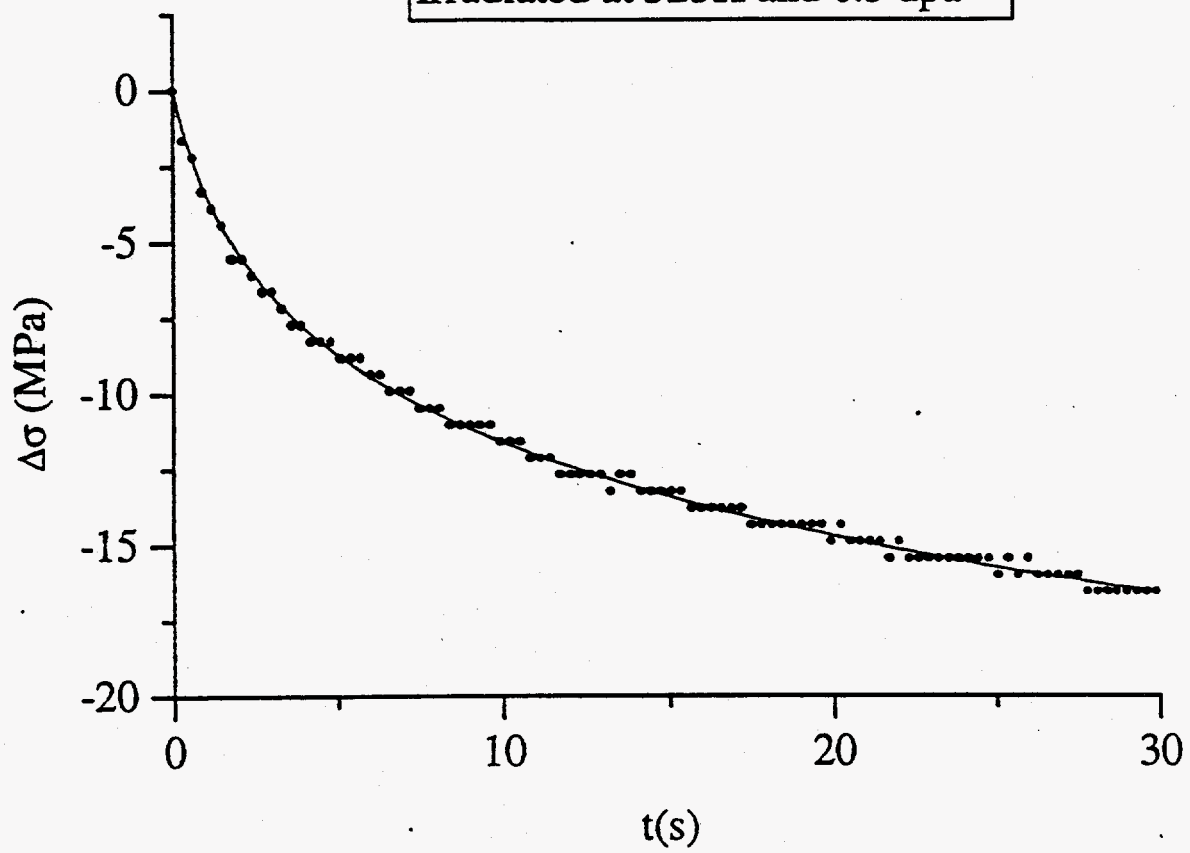
Repeated relaxations:

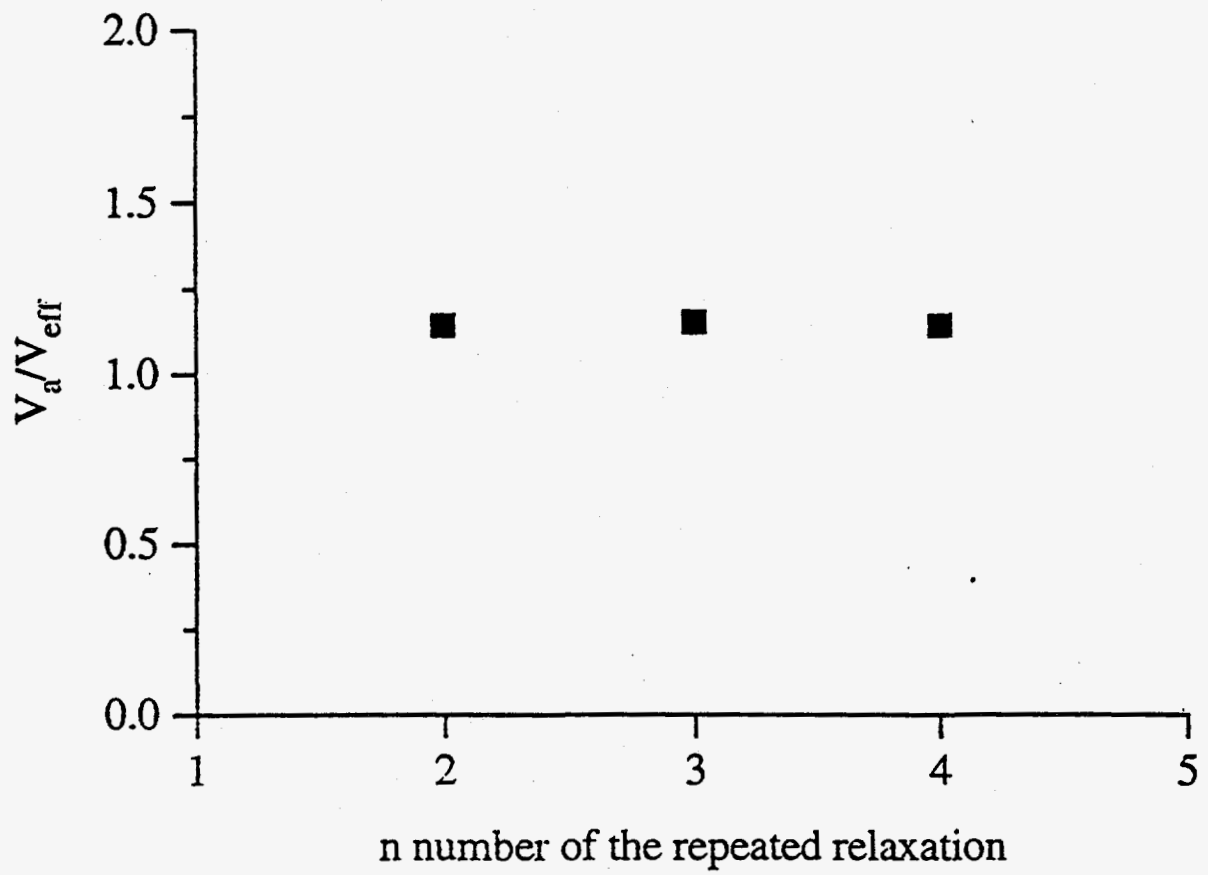
$$\Omega = \frac{V_a}{V_{\text{eff}}} = \left(1 + \frac{K}{M}\right)(1 + \beta) = \left(1 - \frac{kT}{V_a \sum_{j=1}^{n-1} \Delta\tau_j} \ln\left(\frac{\exp(-\Delta\tau_n V_a/kT) - 1}{\exp(-\Delta\tau_1 V_a/kT) - 1}\right)\right)^{-1}$$

# Example of stress relaxation test at Y.S

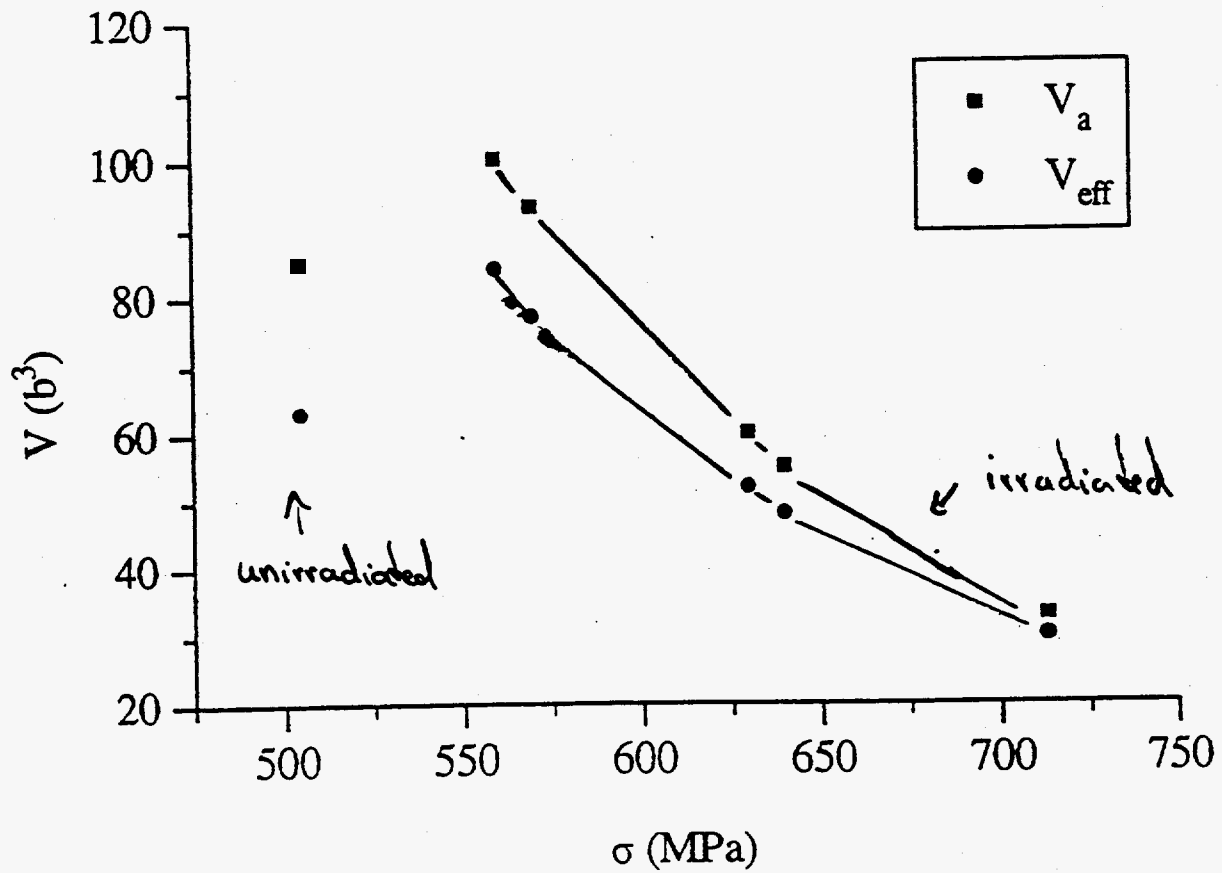


Irradiated at 523K and 0.3 dpa





# Stress dependence of the activation volume



## Summary - future task

- First measurements of the radiation hardening on specimens irradiated in Pirex at 0.3 and 0.5 dpa
- Activation volume measurements have been performed and must be completed
- Irradiation of the same PIREX specimens is in progress in ~~the~~ Studvik reactor on the preirradiated specimen in PIREX and on virgin specimens
- PIREX irradiation at higher doses (up to 2 dpa) have been done this year

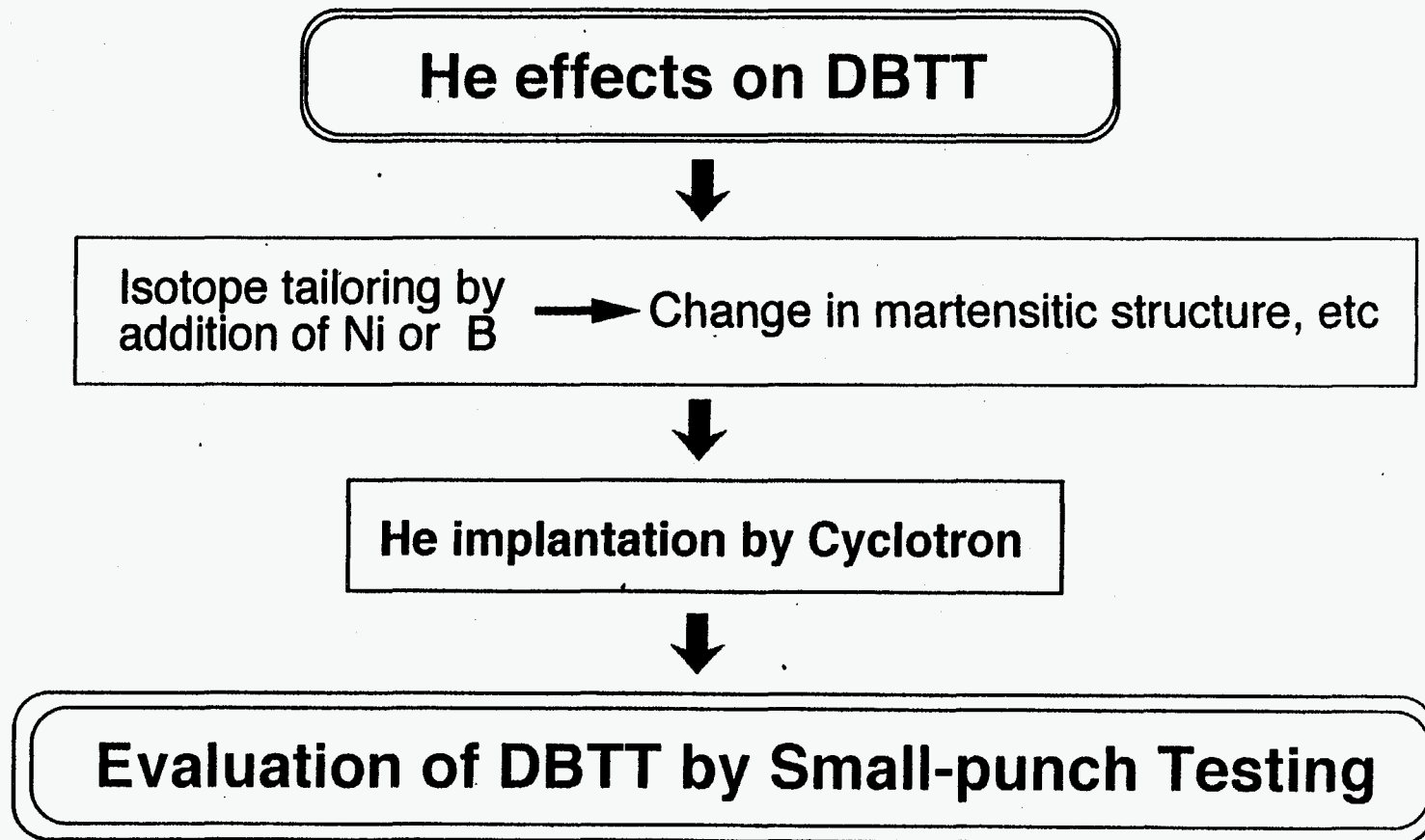
The IEA Working Group meeting  
on 24-25 October 1996  
Culham Laboratories at Abingdon

# Effect of He-implantation on DBTT of Reduced Activation Martensitic Steels

A. Kimura, T. Morimura, H. Matsui

Institute for Materials Research,  
Tohoku University





# Chemical composition

## ○Chemical composition (mass%) : 9%Cr-2%W steel

	C	Si	Mn	P	S	Cr	W	V	Ta	Ti	B	Y	Al
NLF-1	0.10	0.042	0.53	0.002	0.0014	9.03	2.06	0.26	0.051	0.021	0.0032	—	—

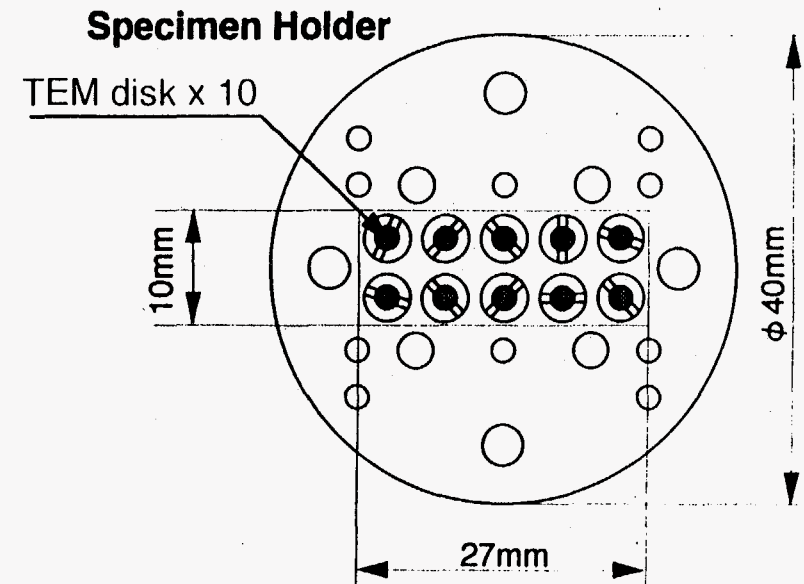
## ○Heat treatment condition

Normalized at 1323K for 30 min and then tempered at 1033K for 30 min, followed by air cooling.

# Experimental

## ○ Cyclotron Irradiation: Homogeneous implantation used by degrader

Incident particle	36MeV- $\alpha$
Implantation temperature	220 $\pm$ 50°C
Total amount of He	120 appm
Displacement damage	0.048dpa
Range	0.23mm
Implantation time	10.5h



## ○ Specimens

$\phi$  3mm TEM disk (0.22mm t) x 10

## ○ Small Punch (SP) test

## ○ Fractography : SEM

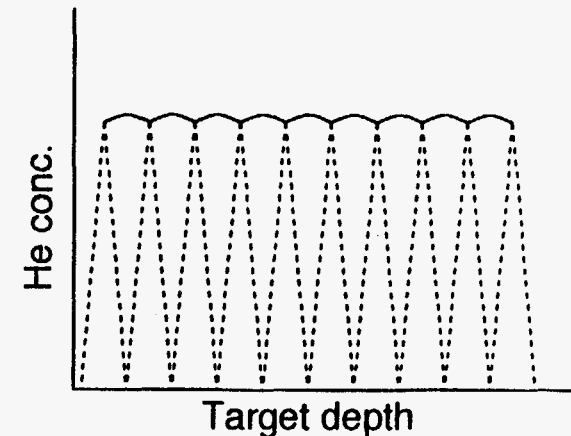
## ○ Observation of Microstructure : TEM

## ○ Positron Annihilation Measurement

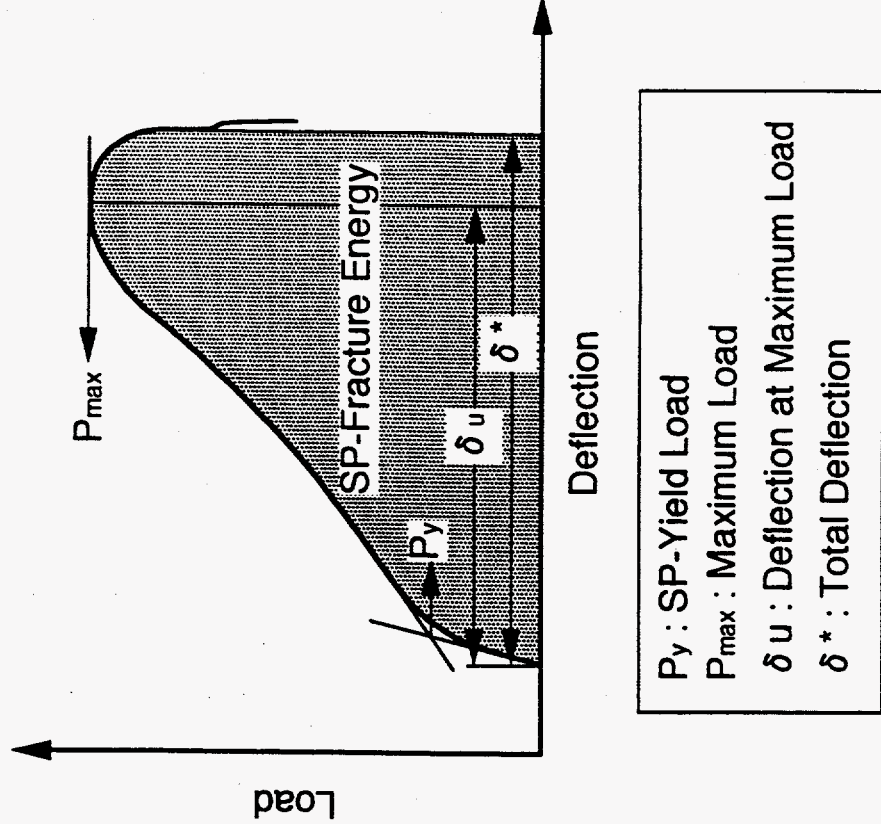
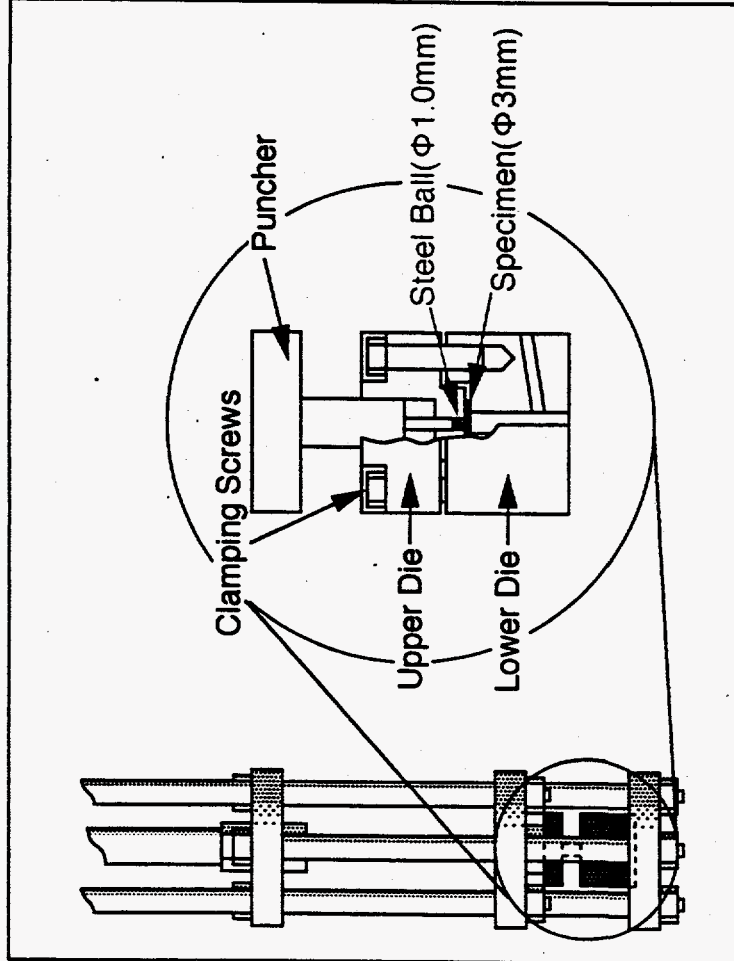
## ○ Post-Implantation Annealing

Annealing condition : 423 ~ 873K - 1h ( $2.0 \times 10^{-5}$  Pa)

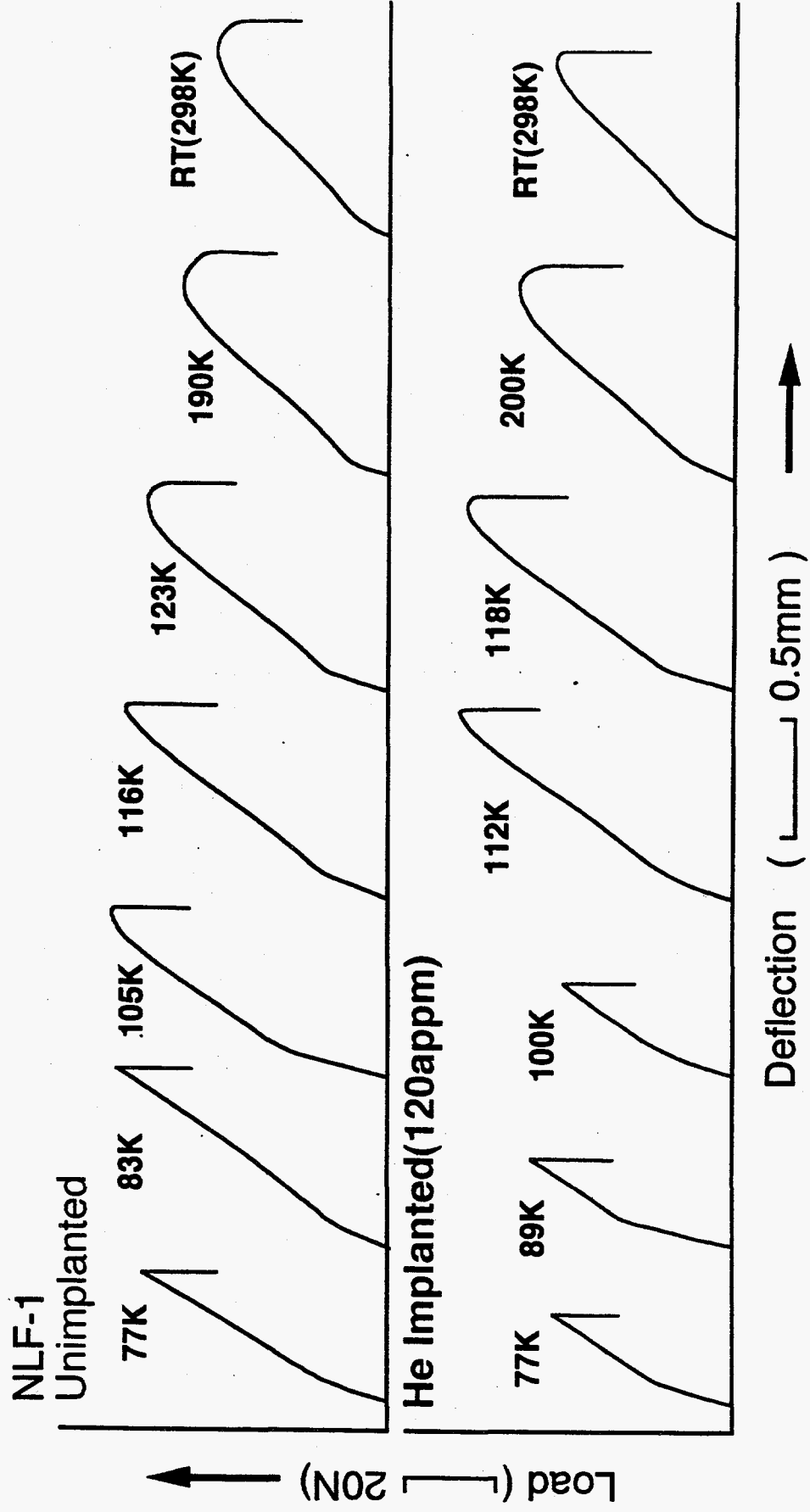
#Vickers hardness measurement (200g)



# Small Punch Test

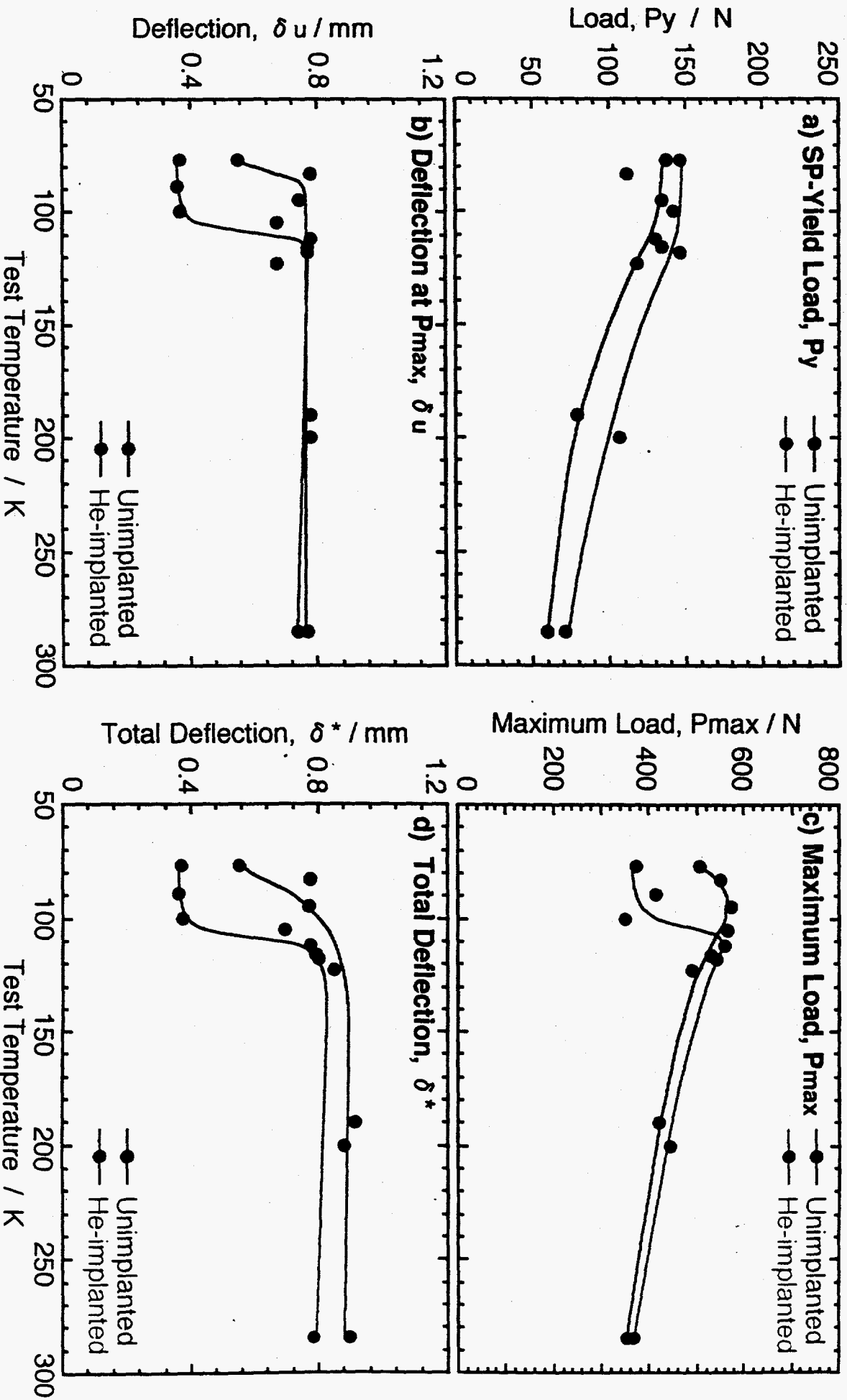


# Load-Deflection curves

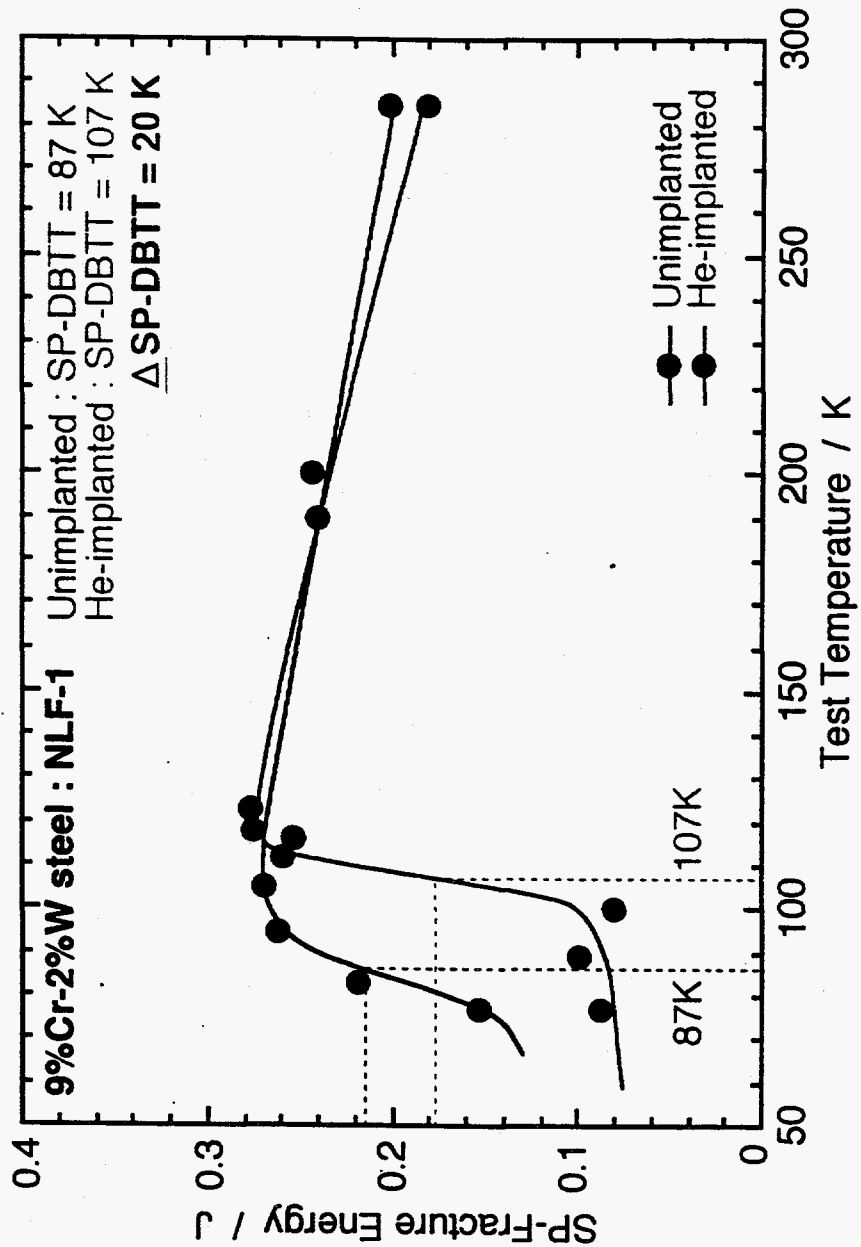


# Results of Small Punch Test

9%Cr-2%W steel : NLF-1

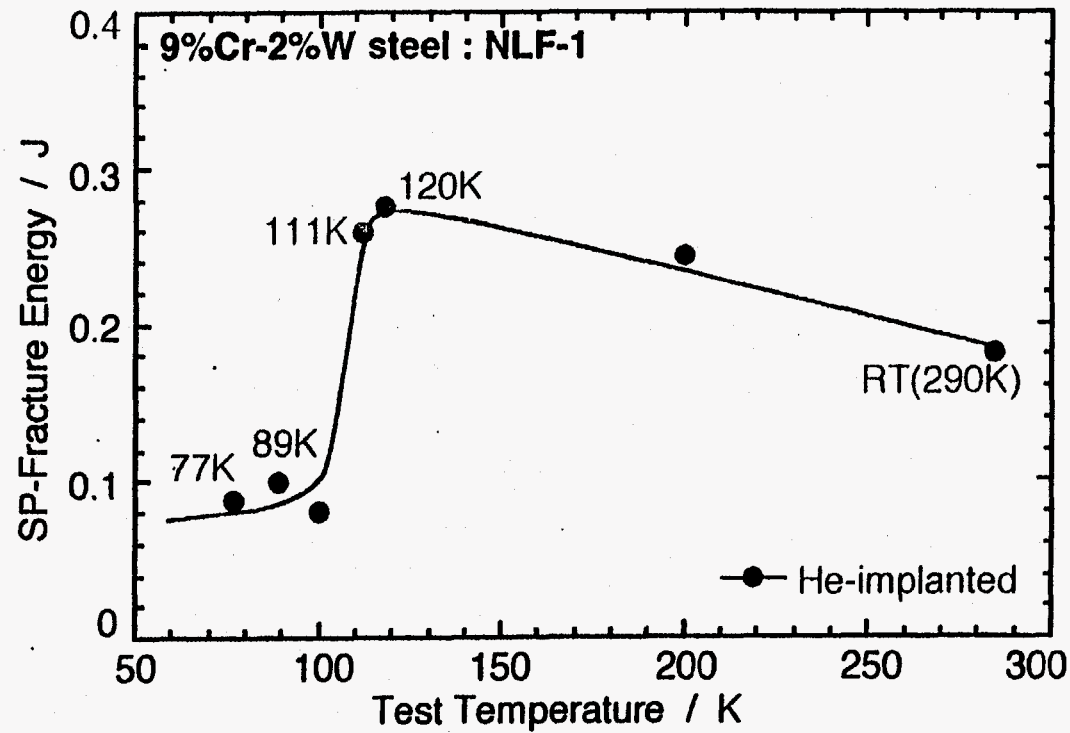


# Determination of SP-DBTT



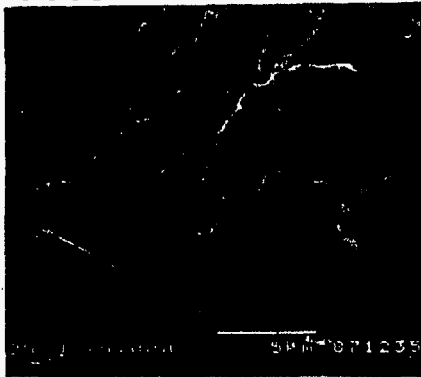
# SEM Observation after Small Punch Test

Institute for Materials Research, TOHOKU UNIVERSITY

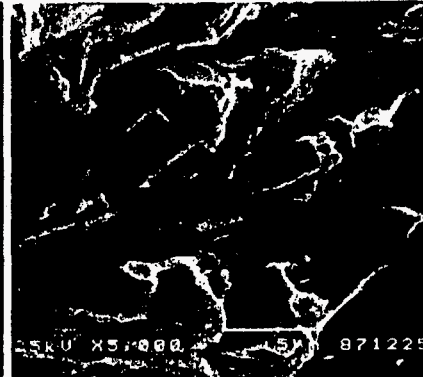


He implanted

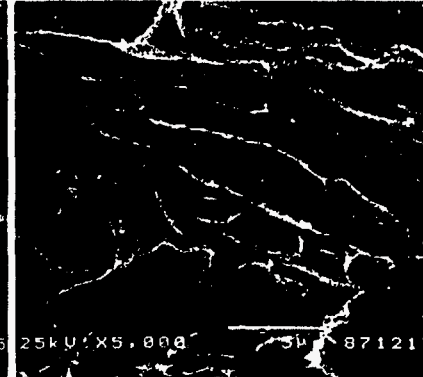
77K



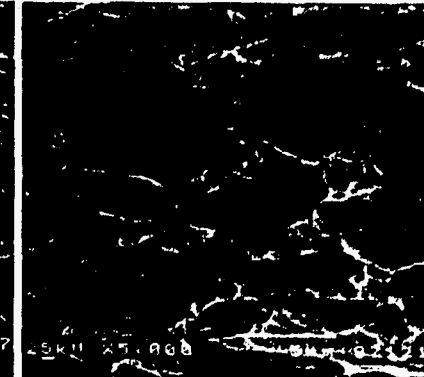
89K



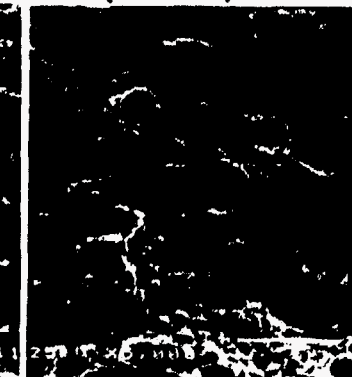
111K



120K



RT(290K)





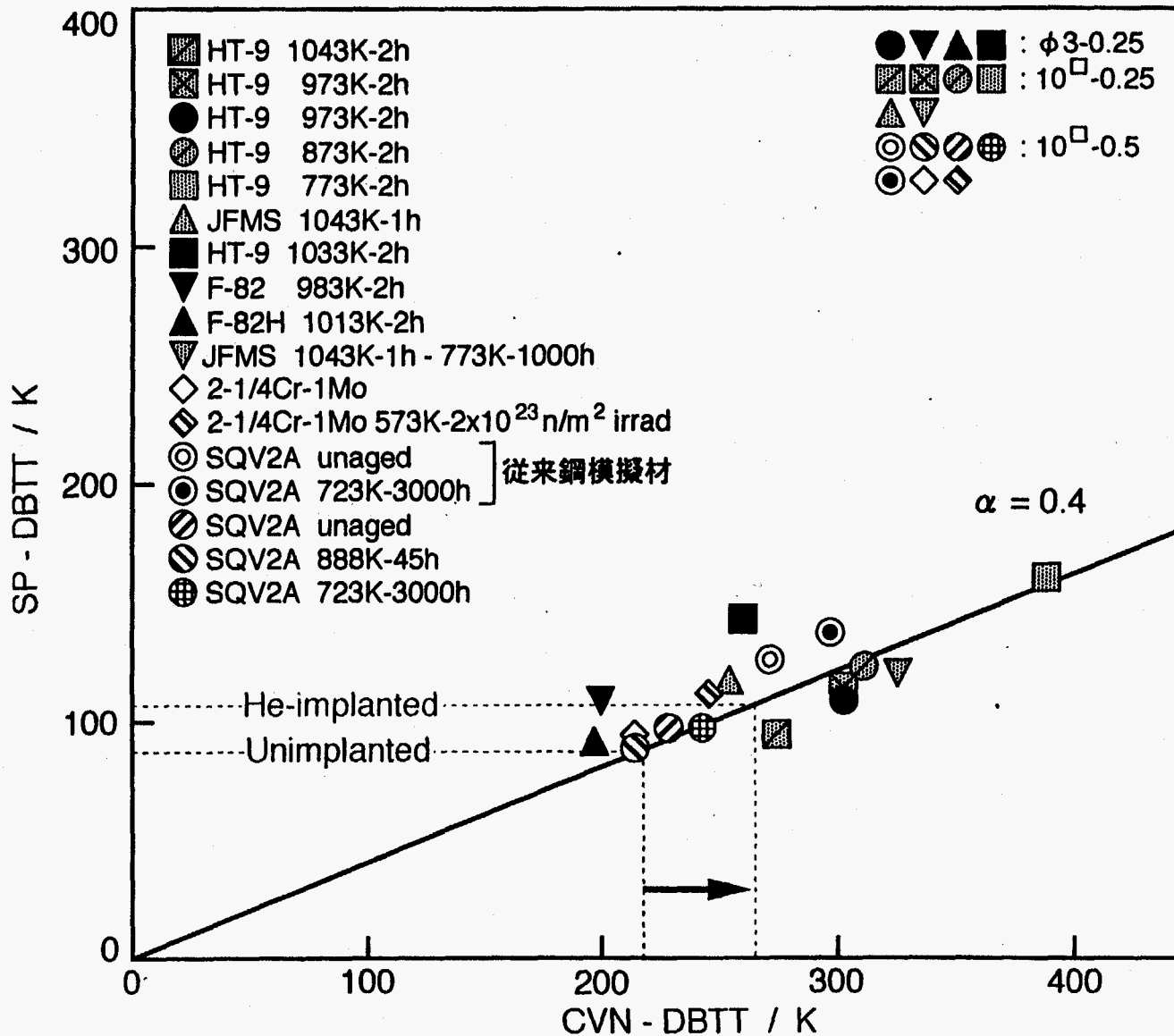
# TEM Observation after He-implantation

Institute for Materials Research, TOHOKU UNIVERSITY

He-implanted



# Estimation of CVN-DBTT (Full-size)



○ Estimation of CVN-DBTT (Full-size)

$$SP-DBTT(K) = \alpha \times CVN-DBTT(K)$$

$$\alpha = 0.4$$

#Unimplanted :  
CVN-DBTT(Full-size) = 218K

#He-implanted :  
CVN-DBTT(Full-size) = 268K

※  $\Delta CVN-DBTT(Full-size) = 50K$

Misawa, Isoo *et al.*  
(Muroran Institute of Technology)

# Correlation between hardness and tensile test

Institute for Materials Research, TOHOKU UNIVERSITY

○  $\sigma_y$  (MPa)  $\propto$  HV

#Unimplanted (RT) :

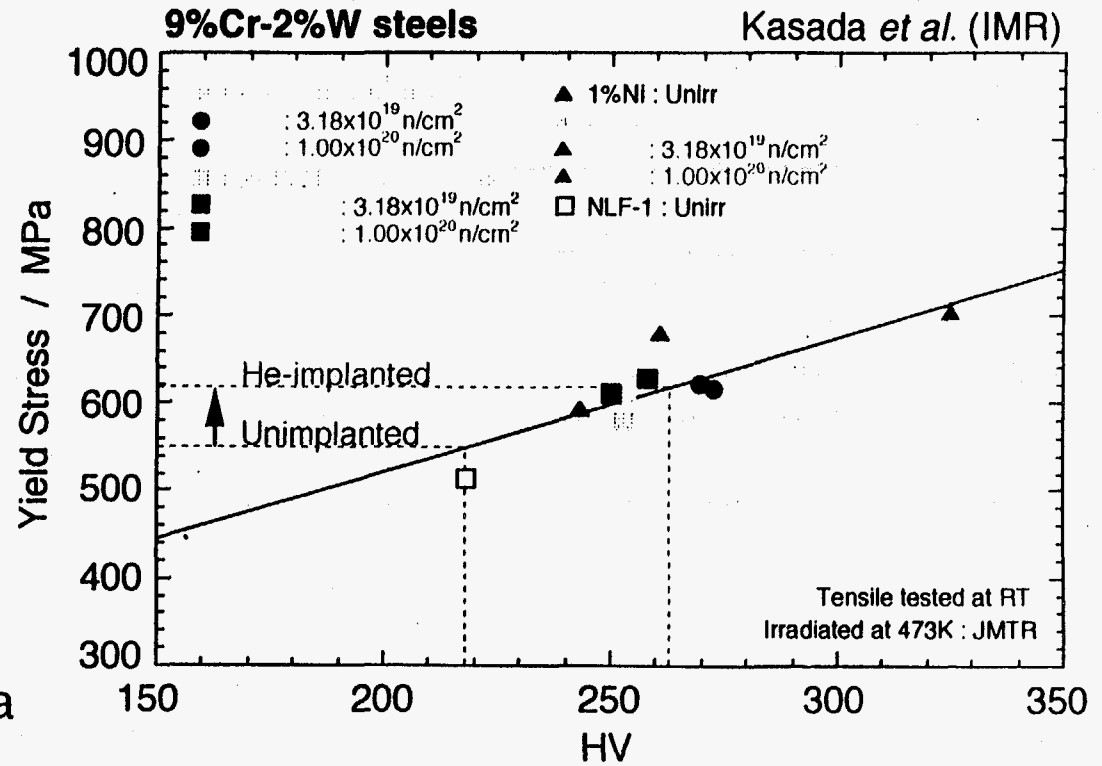
$$\left[ \begin{array}{l} \text{HV} = 218 \text{ (Experimental)} \\ \sigma_y = 514 \text{ MPa (Calculation)} \end{array} \right.$$

$$\sigma_y \text{ (MPa)} = 1.54 \times \text{HV} + 213$$

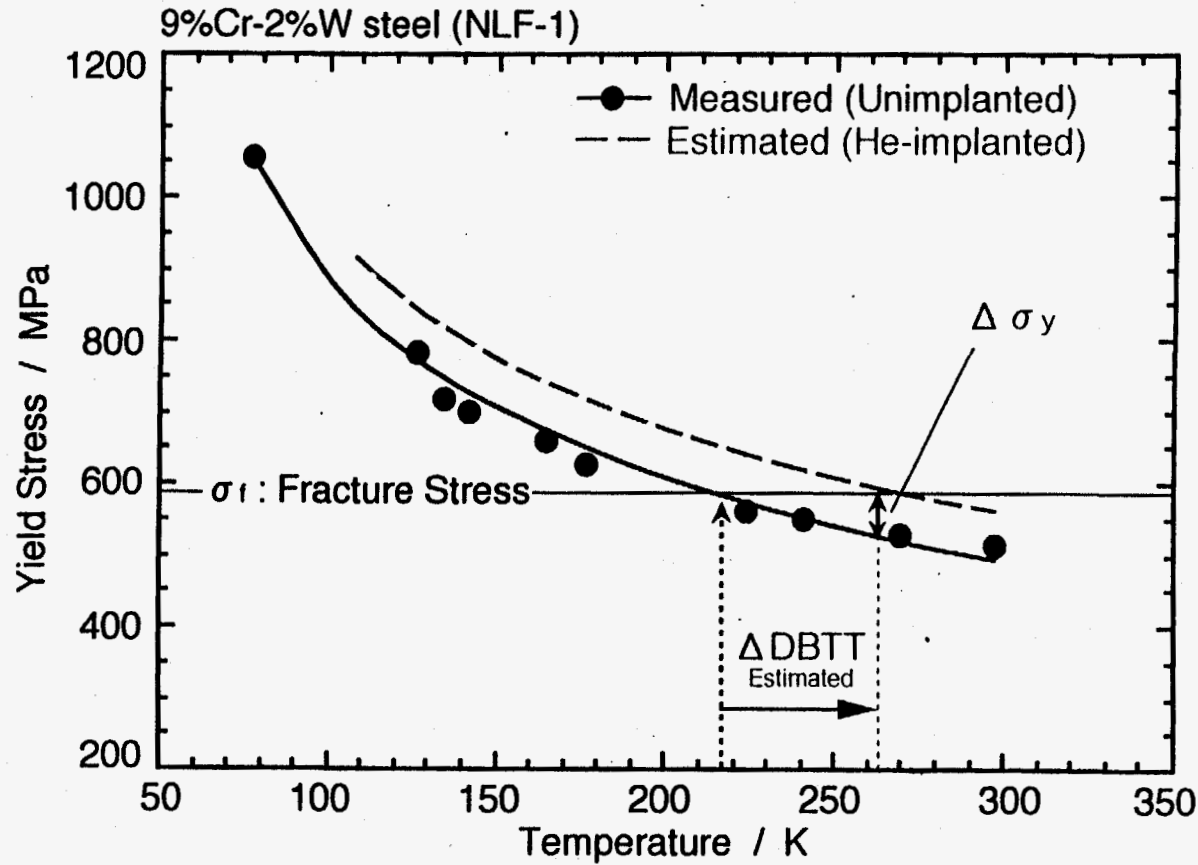
#He-implanted (RT) :

$$\left[ \begin{array}{l} \text{HV} = 263 \text{ (Experimental)} \\ \sigma_y = 618 \text{ MPa (Calculation)} \end{array} \right.$$

$$\text{※ } \Delta \sigma_y \text{ (RT)} = 618 - 514 = 104 \text{ MPa}$$



# Shift in DBTT by He-implantation

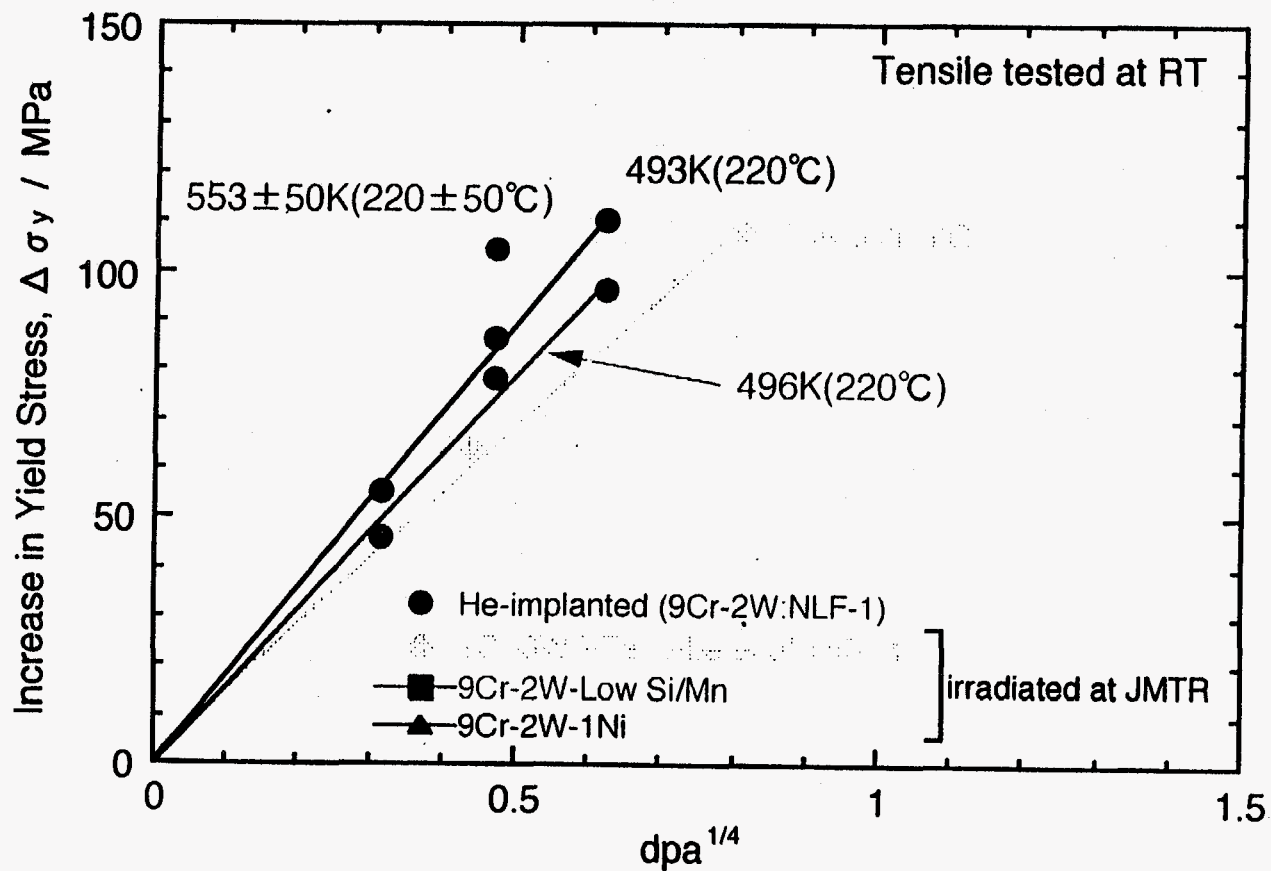


$$\Delta \sigma_y \text{ (Estimated)} \\ = 80 \text{ MPa}$$

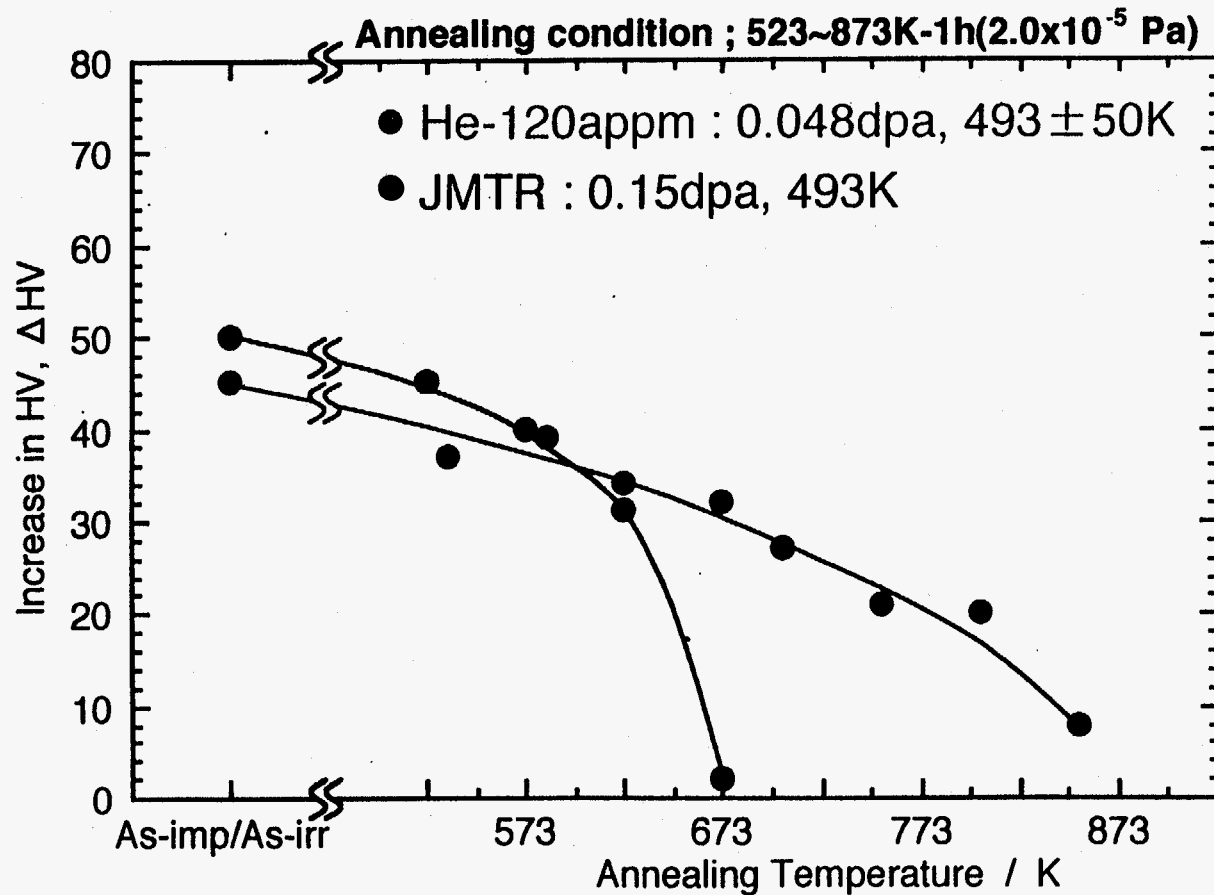


#No decrease  
in Fracture Stress

# Dependence of $\Delta \sigma_y$ on Displacement Damage **IMR** *Tohoku University*



# Hardness change by Post-Implantation Annealing



## Summary

- He-implantation (120appm, 0.048dpa,  $220 \pm 50^\circ\text{C}$ )
  - $\Delta \text{SP-DBTT} = 20 \text{ K}$   $\rightarrow$   $\Delta \text{CVN-DBTT (Full-size)} = 50\text{K}$
  - $\Delta \text{HV} = 45$   $\rightarrow$   $\Delta \sigma_y = 104 \text{ MPa}$
- SEM observation after SP test (Cleavage mode)  $\rightarrow$  No Intergranular Embrittlement caused by He  
TEM observation after He implantation
- Increase in DBTT by He-implantation  $\rightarrow$  Due to Increase in Yield Stress (Not observed decrease in Fracture Stress)
- Implantation temperature  $> 220^\circ\text{C}$   $\rightarrow$  Possibility of Enhanced Hardening by He
- Retardation of recovery after post-implantation annealing
  - $\rightarrow$  Increase in Thermal Stability of Radiation Defect Clusters (retardation of recovery of martensitic structure---high heat resistance)



*IEA Workshop and Working Group Meeting  
on Ferritic Steels  
JET Headquarter, Culham, UK  
24-25 October, 1996*

## **Recent Results on the Second Heat of JLF-1**

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## Progress of Test Program of JLF-1 Steel

Table 1 Test items of first and second heat of JLF-1 steels.

Heat	Test Items
<b>First Heat</b> : 1ton and 300kg small heats 15 and 7mm thickness	<ul style="list-style-type: none"><li>• Mechanical properties of steels and welds (unirradiated and irradiated)</li><li>• Thermal stability of steels and welds (unirradiated and irradiated)</li><li>• Composition optimization</li></ul>
<b>Second Heat</b> : 1.5ton heat 25 and 15mm thickness	<ul style="list-style-type: none"><li>• Fracture toughness (influence of irradiation and magnetic field)</li><li>• Further investigation on influence of neutron irradiation on mechanical properties thermal stability of steels and welds</li><li>• Welding technologies (TIG and EB welding)</li></ul>

Table 2 JLF1-HEAT2 Fabrication Data

Melting	300kg VIM Furnace (0.1C-0.05Si-0.45Mn-9Cr-2W-0.2V-0.08Ta-0.025N)
Casting	300kg Ingot(240tx240wx640h*)x5 *Except hot top
Forging*	Homogenizing : 1250°Cx1h Forging to 100tx330wx1100h *After cutting hot top
Slab Cutting	100tx330wx1100h → 100tx330wx550h (for 15mmt plate) x5slabs → 100tx330wx550h (for 25mmt plate) x5slabs
Hot Rolling	Homogenizing : 1250°Cx3h ①Reduction to 15mmt ②Reduction to 25mmt
Plate Cutting	①15tx330wx450h 25 plates ②25tx330wx500h 15 plates (Saw Cut)
Normalizing	1050°Cx1h/A. C.
Tempering	780°Cx1h/A. C.
PWHT	740°Cx3h/F. C.

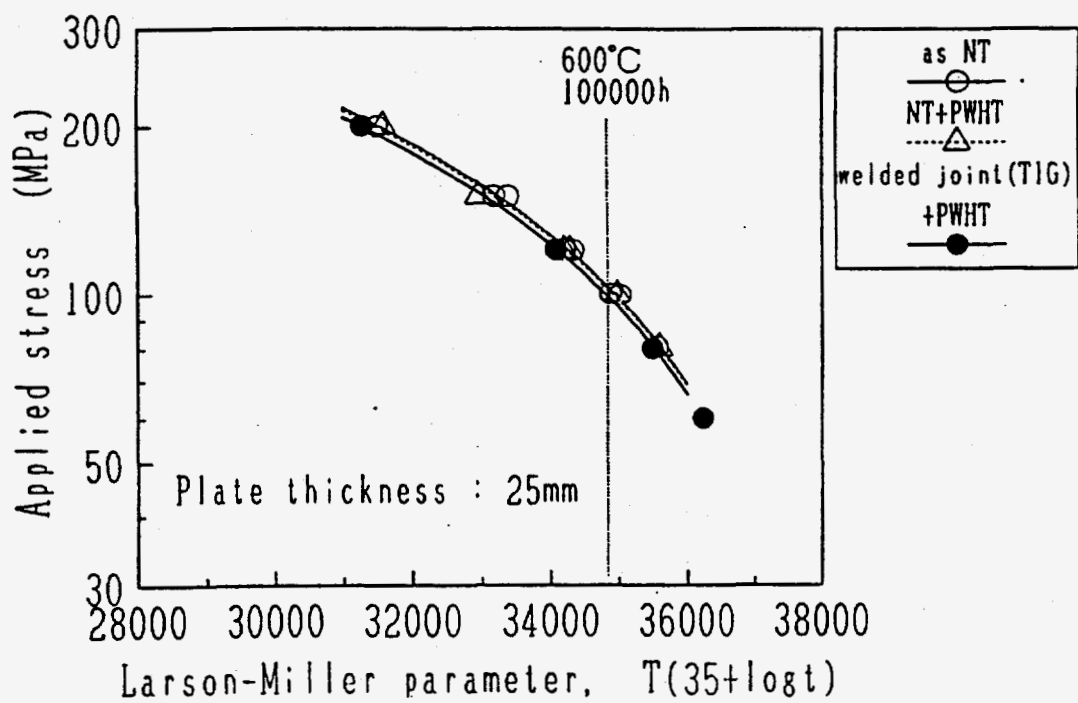
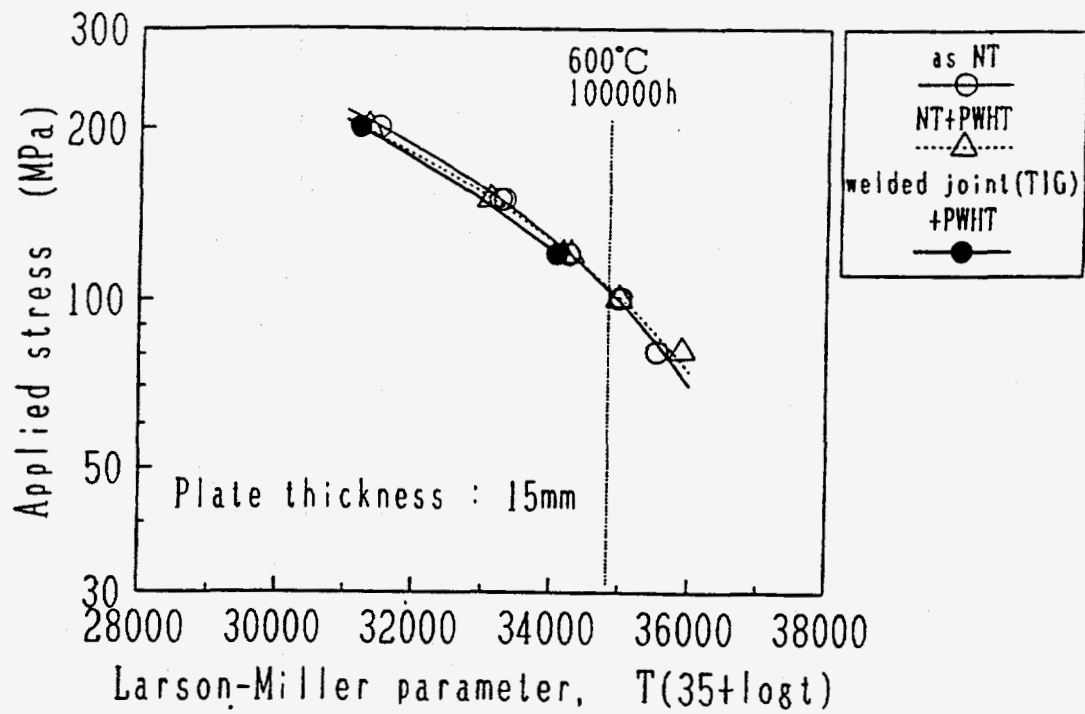


Fig.2 Creep rupture strength of JLF1-HEAT2 steel and its TIG-weldments.

Baseline Properties : JLF1-HEAT2 Steel

Table 3 Chemical compositions of JLF1-HEAT2 steel. (mass%)

	C	Si	Mn	P	S	T-Al	Cr	W	V	Ta	T-N	T-B	Ni	Cu	Mo	Nb
Check analysis	0.10	0.05	0.45	0.003	0.002	0.003	8.85	1.99	0.20	0.080	0.0231	0.0002	<0.01	<0.05	<0.001	<0.002
Targetted value	0.10	0.05	0.45	≤0.005	≤0.005	ALAP	9.00	2.00	0.20	0.080	0.0250	≤0.0010	ALAP	ALAP	ALAP	ALAP

Table 4 Concept for determining chemical compositions

Element	Optimum concentration	Notice
C	0.07 ~ 0.1%	<ul style="list-style-type: none"> <li>• Less C : Tends to <math>\delta</math>-ferrite <math>\rightarrow</math> decrease of toughness</li> <li>• Excess C : decrease of toughness in steels and welds degradation of weldability</li> </ul>
V	0.2%	<ul style="list-style-type: none"> <li>• Improves creep strength</li> <li>• Excess V addition tends to cause deterioration of creep strength and toughness</li> </ul>
Cr	7~9%	<ul style="list-style-type: none"> <li>• Less and Excess Cr decreases stability of microstructure and strength during neutron irradiation</li> </ul>
Ta	0.07 ~ 0.1%	<ul style="list-style-type: none"> <li>• Improves creep strength</li> <li>• Improves toughness ( presumably due to grain refinement )</li> <li>• Excess Ta tends to cause deterioration of toughness</li> </ul>
N	to be determined	<ul style="list-style-type: none"> <li>• Improves high temperature creep strength</li> <li>• Less N is desirable from low activation consideration</li> </ul>

Photo. 1 Optical microstructure of JF1+EA2 steel.

Plate thickness	Heat treatment	0.1mm	0.02mm
15mm	as NT		
	NT+PWHT		
25mm	as NT		
	NT+PWHT		

Table 5 Tensile properties of JLF1-HEAT2 steel at room temperature.

Plate thickness	Heat treatment	Direction	0.2%P.S. (MPa)	T.S. (MPa)	T-EL (%)	R.A (%)
15mm	as NT	Longitudinal	466	631	24.7	77.9
		Transverse	474	631	25.4	76.6
	NT+PWHT	Longitudinal	447	617	27.1	75.8
		Transverse	445	613	26.4	75.9
25mm	as NT	Longitudinal	457	630	28.0	76.4
		Transverse	469	634	26.2	74.2
	NT+PWHT	Longitudinal	456	630	27.2	74.8
		Transverse	455	624	25.7	71.8

Table 6 Tensile properties of JLF1-HEAT2 steel at 600°C.

Plate thickness	Heat treatment	Direction	0.2%P.S. (MPa)	T.S. (MPa)	T-EL (%)	R.A (%)
15mm	as NT	Longitudinal	253	315	24.9	89.3
		Transverse	259	317	23.7	88.7
	NT+PWHT	Longitudinal	243	304	26.3	90.9
		Transverse	243	301	25.7	90.3
25mm	as NT	Longitudinal	252	313	26.0	89.2
		Transverse	253	316	28.6	90.2
	NT+PWHT	Longitudinal	247	313	28.5	89.1
		Transverse	242	310	27.1	90.0

Table 7 Charpy impact test results of JLF1-HEAT2 steel.

Plate thickness	Heat treatment	Direction	Test temp. (°C)	Absorbed energy (J)				FATT (°C)
				1	2	3	ave.	
15mm	as NT	Longitudinal	0	262	228	262	251	-37
			-40	171	60	142	124	
			-60	17	145	12	58	
		Transverse	0	259	232	234	242	
			-40	177	82	38	99	
			-60	49	21	26	32	
	NT+PWHT	Longitudinal	0	214	260	214	229	-29
			-40	51	158	64	91	
			-60	29	17	9	18	
		Transverse	0	239	264	290	264	
			-40	173	89	143	135	
			-60	34	32	17	28	
25mm	as NT	Longitudinal	0	268	267	271	269	-55
			-40	242	235	257	245	
			-60	219	94	156	156	
		Transverse	0	259	257	276	264	
			-40	243	216	191	217	
			-60	174	116	121	137	
	NT+PWHT	Longitudinal	0	255	262	249	255	<-60
			-40	225	234	239	233	
			-60	196	165	227	196	
		Transverse	0	257	257	254	256	
			-40	249	246	252	249	
			-60	199	53	21	91	



## Baseline Properties : TIG welds of JLF1-HEAT2 Steel

Table 9 TIG welding conditions.

Plate thickness	15mm	25mm
Current	230~250A	
Voltage	10.5V	
Travel speed	10cm/min.	
Heat input	14.5~15.8kJ/cm	
Preheat temp.	≤200°C	
Interlayer temp.	≤200°C	
Number of passes	≐8	≐20
Wire diameter	1.2mm	
PWHT condition	740°Cx3h/FC	

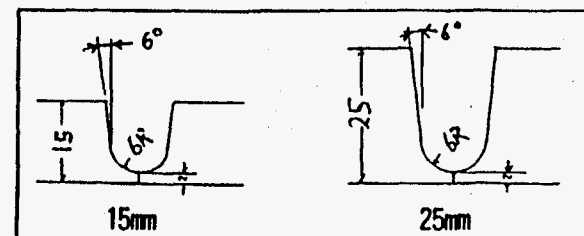


Table 10 Chemical compositions of welding wire. (mass%)

C	Si	Mn	P	S	T-Al	Cr	W	V	Ta	Ti	T-N	T-B
0.061	0.10	0.45	0.003	0.003	0.002	8.96	1.82	0.25	0.084	0.028	0.0332	0.0001

Table 11 Chemical compositions of weld metal. (mass%)

Plate thickness	C	Si	Mn	P	S	T-Al	Cr	W	V	Ta	Ti	T-N	T-B
15mm	0.066	0.13	0.43	0.004	0.003	0.003	9.10	1.85	0.25	0.081	0.018	0.0263	0.0001
25mm	0.061	0.13	0.43	0.005	0.003	0.003	9.16	1.91	0.25	0.081	0.019	0.0259	0.0001

Table 8 Creep rupture test results of JLF1-HEAT2 steel.

Plate thickness	Heat treatment	TP No.	Temp. (°C)	Applied stress (MPa)	Time to rupture (h)	Ei (%)	R. A (%)
15mm	as NT	1	600	200.05	11.1	32.7	88.8
		2		150.04	1332.9	23.1	86.5
		3		120.86			
		4	650	150.06	9.9	34.1	88.4
		5		120.04	126.6	35.0	91.3
		6		100.71	846.3	27.5	90.3
		7	700	120.04	1.6	40.6	93.8
		8		100.06	9.0	51.0	93.7
		9		80.60	33.3	26.3	88.5
	NT+PWHT	600	1	200.05	7.3	37.7	90.1
			2	150.05	451.5	26.3	88.5
			3				
		4	650	150.06	7.1	34.5	90.7
		5		120.04	102.4	34.4	91.8
		6					
		7	700	120.04	1.7	43.3	93.7
		8		100.06	9.0	38.8	94.1
		9		80.84	78.0	42.6	93.5
25mm	as NT	1	600	200.05	12.1	34.7	87.0
		2		150.06	1747.9	26.7	80.3
		3		121.26			
		4	650	150.06	8.9	32.9	92.2
		5		120.04	113.4	32.4	91.8
		6		100.38	605.9	26.8	90.8
		7	700	120.06	1.9	46.3	93.3
		8		100.05	9.6	47.2	93.5
		9		80.60	35.0	42.2	93.4
	NT+PWHT	600	1	200.05	15.3	37.0	89.4
			2	150.05			
			3				
		4	650	150.06	5.1	36.9	91.0
		5		120.04	110.5	32.4	91.3
		6		100.38			
		7	700	120.05	1.7	44.9	93.4
		8		100.06	8.7	43.4	91.9
		9		80.33	38.1	65.3	92.3

Photo. 2 Macrostructure of cross sections of TIG-welded joints.

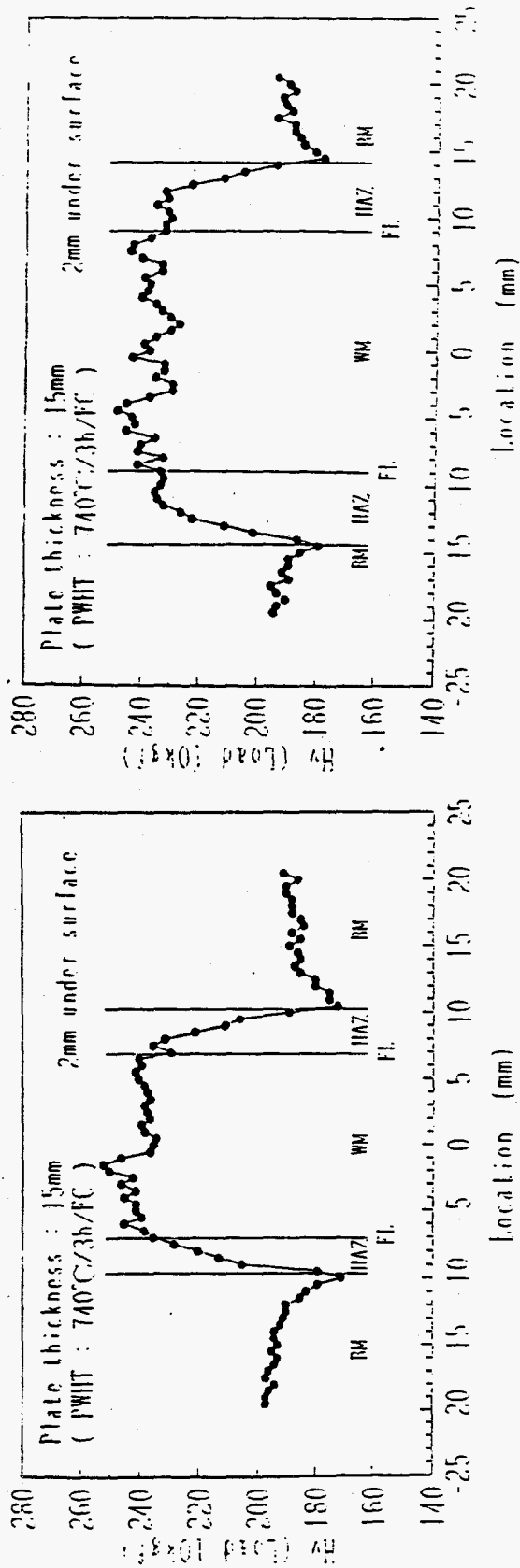
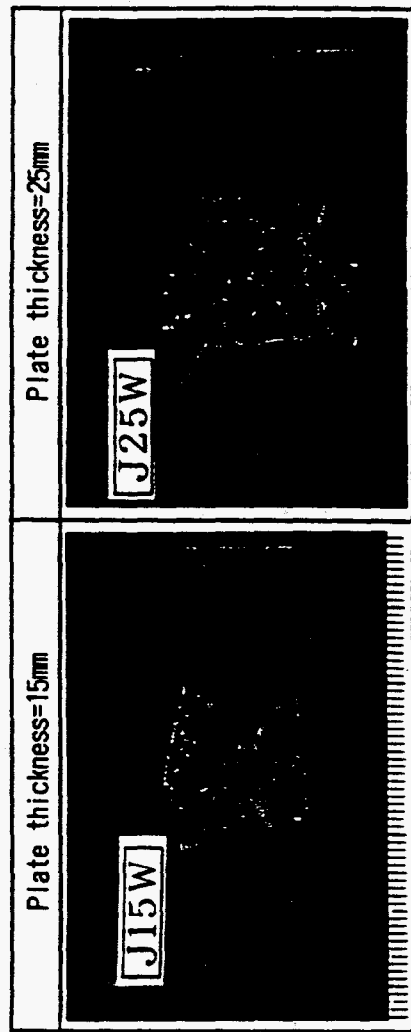


Fig. 1 Hardness distribution of TIG welds.

Photo. 3 Cross sectional microstructure of TIG-welded joints (Plate thickness : 15mm).

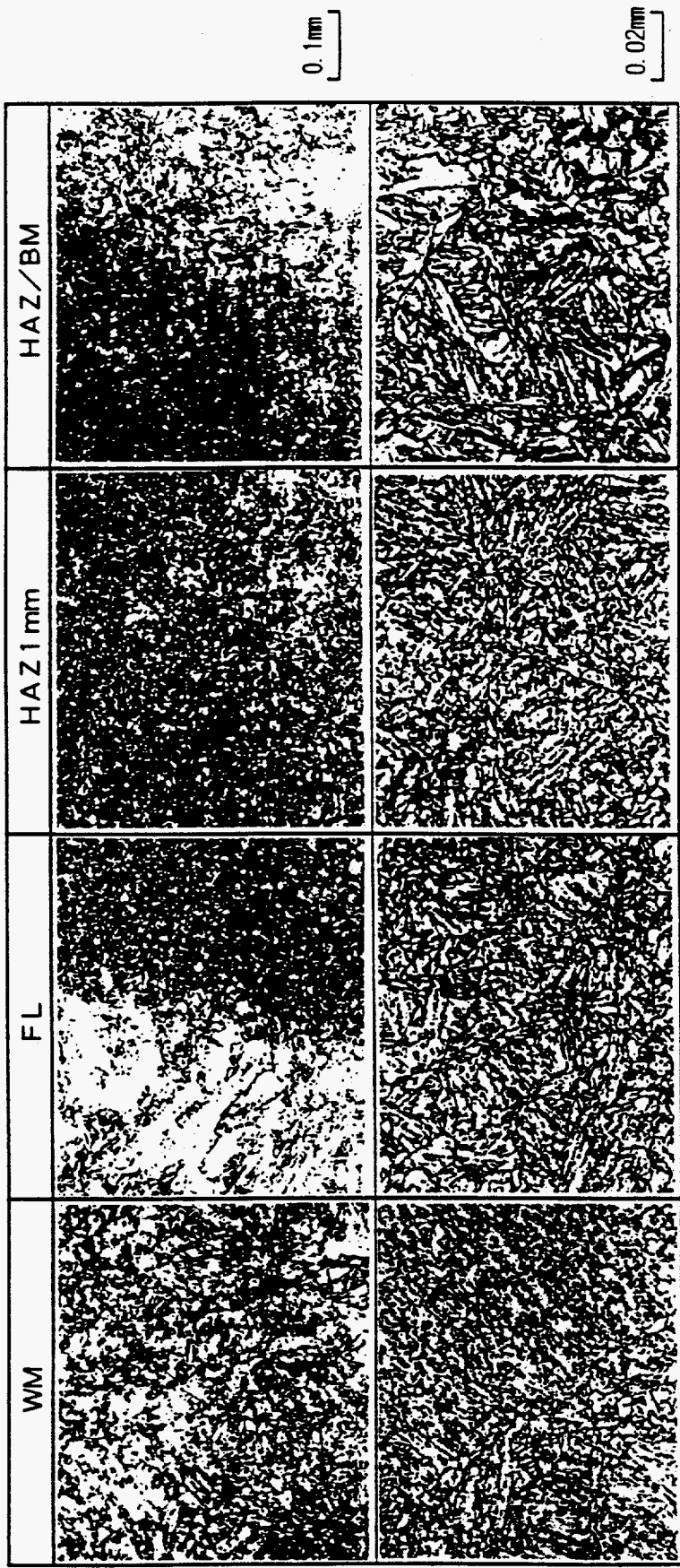
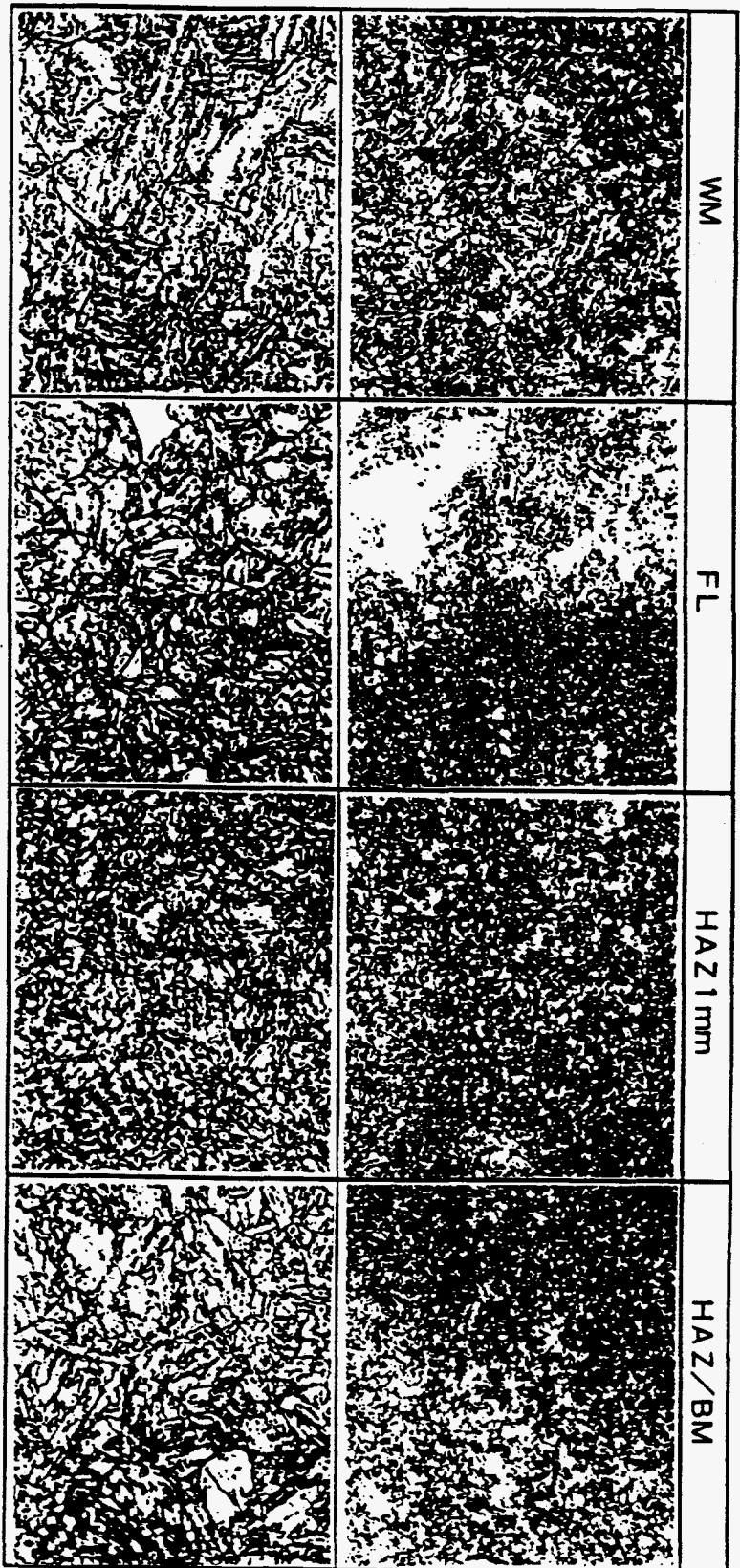


Photo. 4 Cross sectional microstructure of TIG-welded joints (Plate thickness : 25mm).



0.1mm

0.02mm

Table 12 Tensile properties of weld metal.

Plate thickness	0.2% P.S. (MPa)	T.S. (MPa)	T-EL (%)	R.A (%)
15mm	656	755	23.7	76.5
25mm	669	759	22.8	76.2

PWHT:740°Cx3h/FC

Table 13 Tensile properties of welded joint.

Plate thickness	Temperature (°C)	0.2% P.S. (MPa)	T.S. (MPa)	T-EL (%)	R.A (%)	Location of fracture
15mm	RT	447	610	18.9	74.8	BM
		448	611	20.1	79.7	BM
	600	250	293	19.3	89.4	BM
		256	294	20.3	89.8	BM
25mm	RT	464	619	21.7	77.3	BM
		466	619	19.6	75.1	BM
	600	256	308	20.2	88.3	BM
		260	307	21.0	87.9	BM

PWHT:740°Cx3h/FC

Table 14 Charpy impact test results at 0°C of welded joint.

Plate thickness	Notch location	Absorbed energy at 0°C (J)	Crystallinity at 0°C (%)	FATT (°C)
15mm	WM	287	0	<0
		199	10	<0
	FL	242	0	<0
		240	0	<0
25mm	HAZ1mm	211	11	<0
		216	0	<0
	WM	60	60	≅0
		63	56	≅0
FL	250	0	<0	
	257	0	<0	
HAZ1mm	196	19	<0	
	209	20	<0	

PWHT:740°Cx3h/FC

Table 15 Creep rupture test results of welded joint.

Plate thickness	TP No	Temp. (°C)	Applied stress (MPa)	Time to rupture (h)	El. (%)	R. A (%)	
15mm	1	600	200.04	5.4	18.4	87.6	
	2		150.04				
	3	650	120.05	82.4	20.6	89.5	
	4						100.05
	5		200.06				
	6						150.06
25mm	1	600	200.06	6.7	18.3	88.3	
	2						1747.9
	3						
	4	650	120.05	83.9	20.4	90.5	
	5						100.05
	6						
	7	700	80.05	30.0	11.2	67.6	
	8						60.05
	9						

PWHT: 740°Cx3h/FC

Table 16 List of JLF1-HEAT2 steels and weldments (Plate thickness 15mm)

	Symbol	Plate size	Welding <sup>*</sup>	Heat treatment	Notice
1	HEAT2-1-1-1	15x340x450mm	BM	as NT	
2	-2	"	TIG	PWHT	
3	-3A	15x340x220mm	BM	PWHT	HEAT2-1-1-3 →Cut
4	-3B	"	EB	as welded	HEAT2-1-1-3 →Cut
5	-4A	"	BM	as NT	HEAT2-1-1-4 →Cut
6	-4B	"	BM	NT+PWHT	HEAT2-1-1-4 →Cut
7	-5	15x340x450mm	BM	as NT	
8	HEAT2-2-1-1	"	BM	as NT	
9	-2	"	BM	NT+PWHT	
10	-3	"	TIG	PWHT	
11	-4	"	TIG	PWHT	
12	-5	"	BM	as NT	
13	HEAT2-3-1-1	"	TIG	PWHT	
14	-2	"	EB	PWHT	
15	-3	"	BM	as NT	
16	-4	"	BM	as NT	
17	-5	"	BM	NT+PWHT	
18	HEAT2-4-1-1	"	BM	NT+PWHT	
19	-2	"	TIG	PWHT	
20	-3	"	TIG	PWHT	
21	-4	"	EB	as welded	
22	-5	"	BM	as NT	
23	HEAT2-5-1-1A	15x340x220mm	BM	NT+PWHT	HEAT2-5-1-1 →Cut
24	HEAT2-5-1-1B	"	TIG	PWHT	HEAT2-5-1-1 →Cut
25	-2	15x340x450mm	BM	as NT	
26	-3	"	TIG	PWHT	
27	-4	"	BM	as NT and NT+PWHT	Already used for mechanical properties
28	-5	"	TIG	PWHT	

\* BM:Base Plate (without welding) TIG:TIG welding EB:Electron beam welding



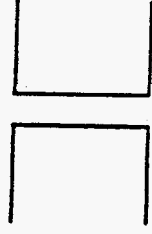
Table 17 List of JLF1-HEAT2 steels and weldments (Plate thickness 25mm)

	Symbol	Plate size	Welding	Heat treatment	Notice
1	HEAT2-1-2-1	25x340x500mm	BM	as NT	
2	-2	"	TIG	PWHT	
3	-3	"	BM	as NT	
4	HEAT2-2-2-1	"	TIG	PWHT	
5	-2	"	BM	as NT	
6	-3	"	BM	NT+PWHT	
7	HEAT2-3-2-1	"	TIG	PWHT	
8	-2	"	BM	as NT	
9	-3	"	BM	NT+PWHT	
10	HEAT2-4-2-1	"	TIG	PWHT	
11	-2	"	TIG	PWHT	
12	-3	"	TIG	PWHT	
13	HEAT2-5-2-1	"	BM	NT+PWHT	
14	-2	"	BM	as NT and NT+PWHT	Already used for mechanical properties
15	-3	"	TIG	PWHT	

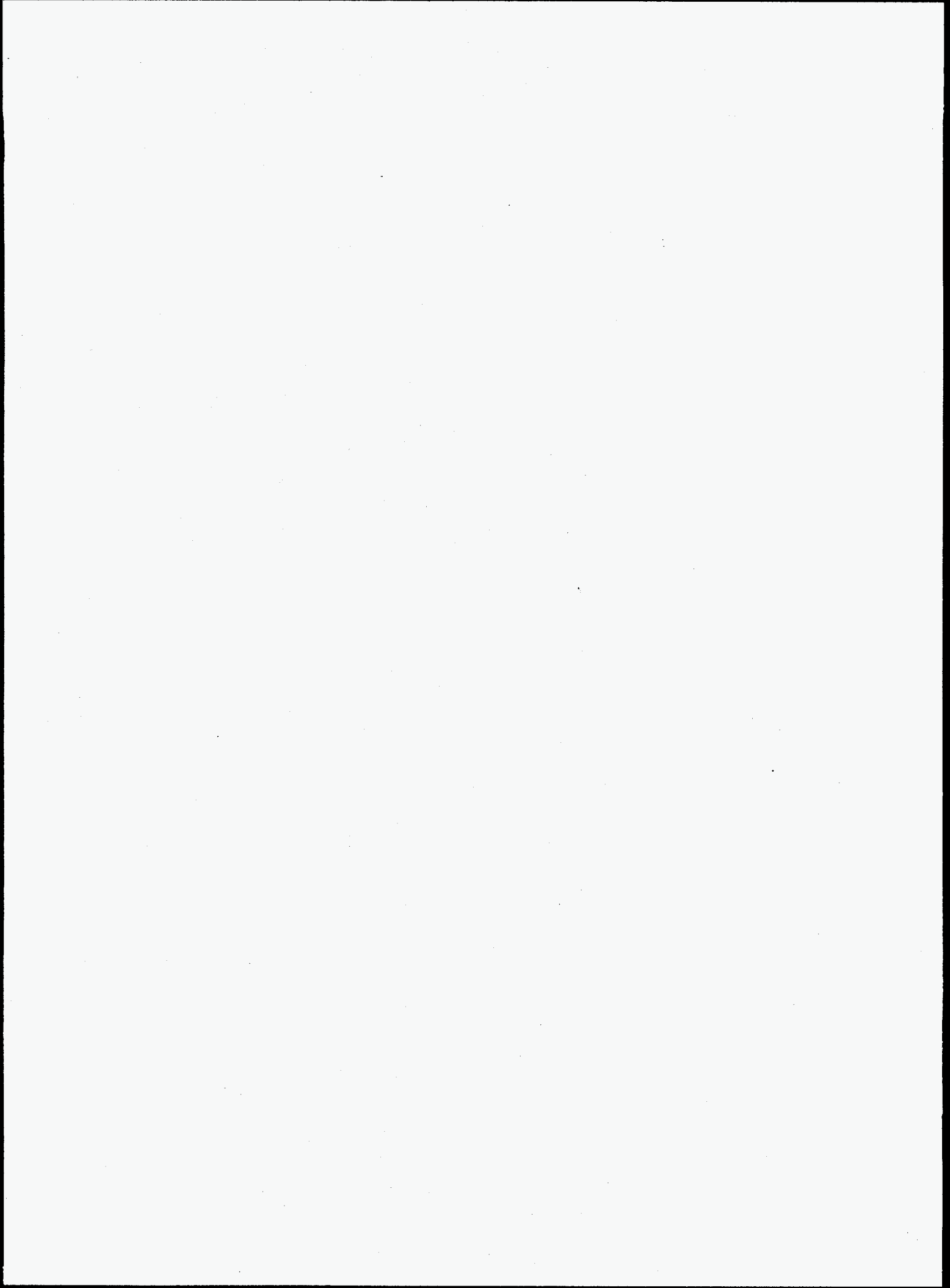
\* BM:Base Plate (without welding) TIG:TIG welding EB:Electron beam welding

Table EB welding conditions.

Plate thickness	15mm
Filament Current	80mA
Acc. Voltage	150kV
Travel speed	50cm/min.
Focus Position	Plate Surface
Preheat	Non
PWHT condition	740°Cx3h/FC



Groove



**IEA Workshop on Ferritic Steels  
Culham, UK, October 24-25, 1996**

**RECENT RESULTS ON IMPACT BEHAVIOR  
OF IRRADIATED LOW ACTIVATION STEELS**

**L. E. Schubert\*  
M. L. Hamilton  
D. S. Gelles**



**Pacific Northwest National Laboratory  
\* Associated Western Universities, UMR**

# Phase I: Low Activation Alloy Development at PNNL

GOAL: TO DETERMINE CHANGE IN IMPACT BEHAVIOR WITH IRRADIATION

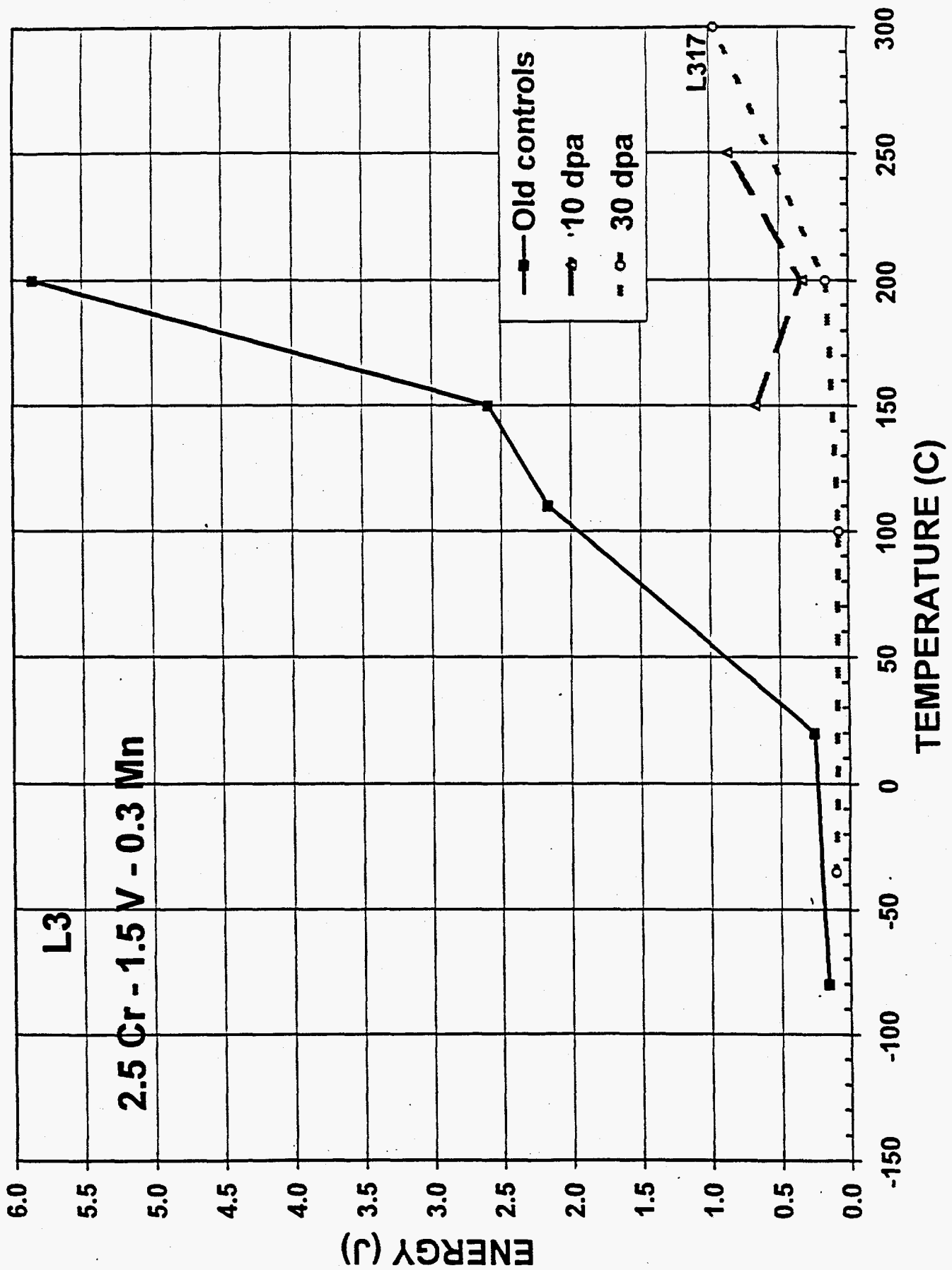
ALLOY DESIGNATION	NOMINAL COMPOSITION
L3	2 Cr-1V
L0 (GA3X)	7.5 Cr-2W
L5	9 Cr-1V-1Mn
L8	9 Cr-1W-2Mn
L7	12 Cr-1V-6Mn
L9	12 Cr-1W-6Mn

# EXPERIMENT DESCRIPTION

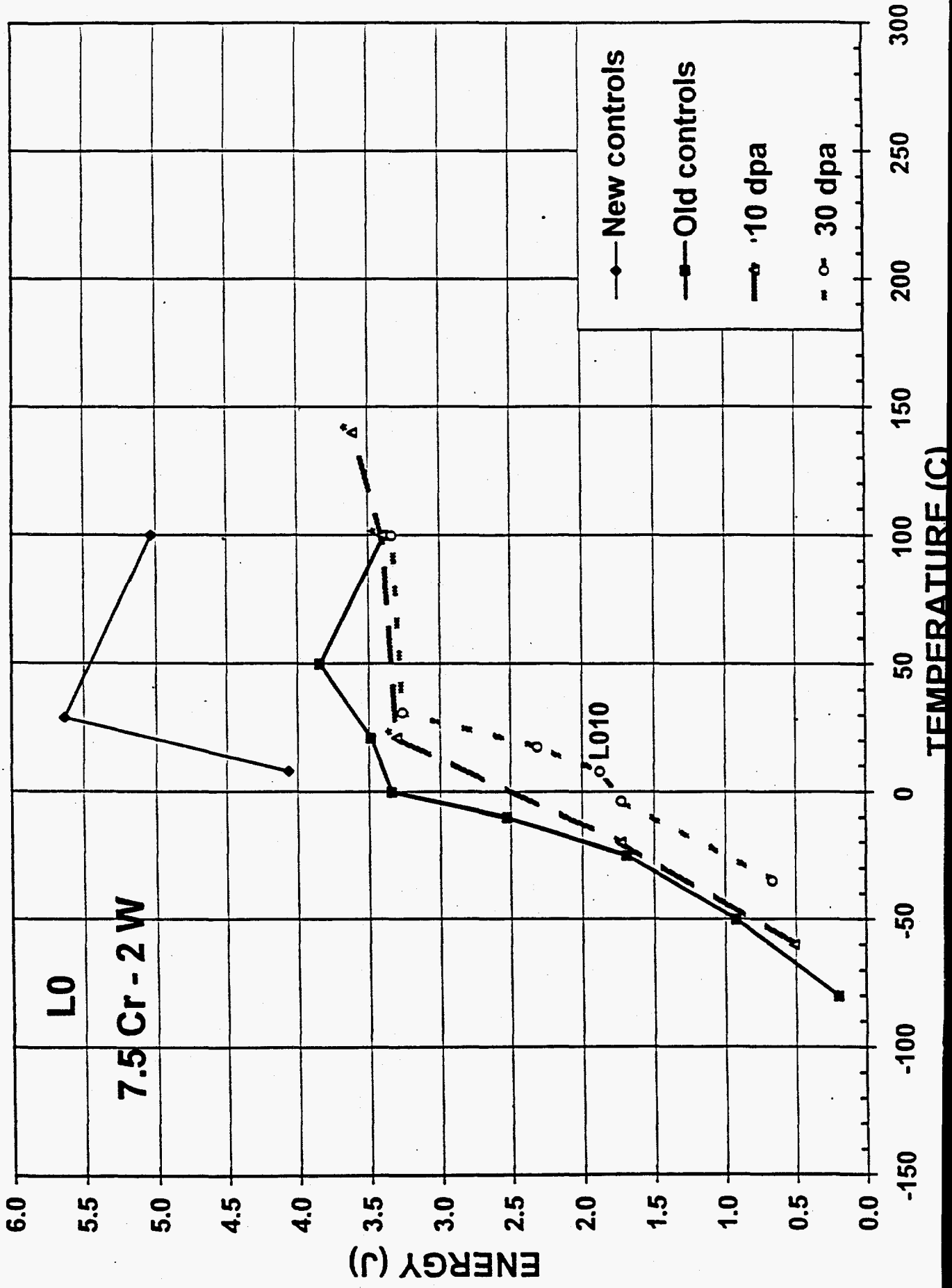
- SPECIMEN SIZE: 1/3 size (3.33 x 3.33 x 23.6 mm)
- SPECIMEN CONDITION: precracked (to  $a/w = 0.4$ )
- IRRADIATION CONDITIONS:
  - in FFTF
  - at  $\sim 370^{\circ}\text{C}$
  - to  $\sim 30$  dpa
- COMPARISON DATA:
  - unirradiated control specimens
  - specimens previously irradiated to  $\sim 10$  dpa

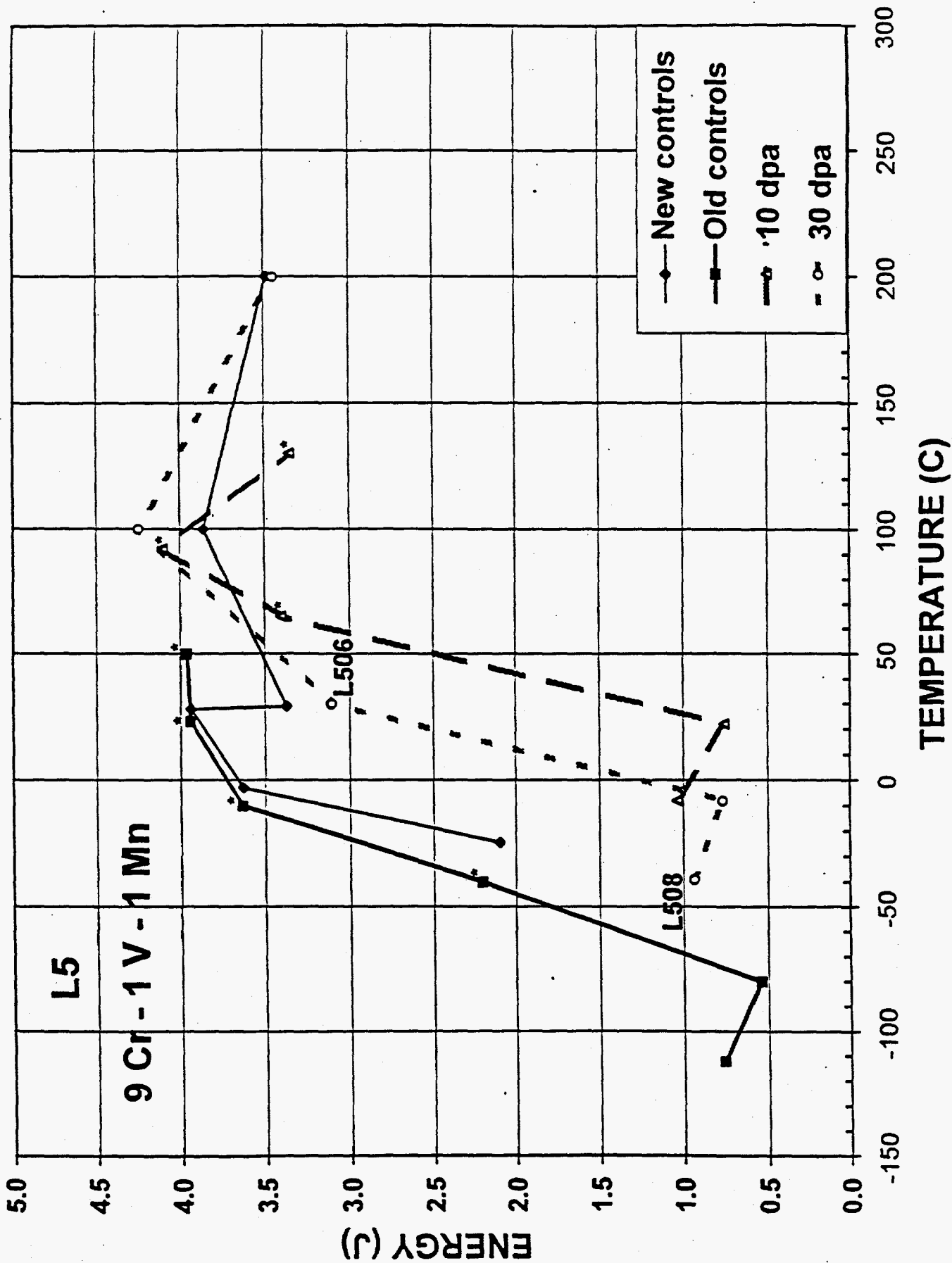
# IMPACT TEST SYSTEM

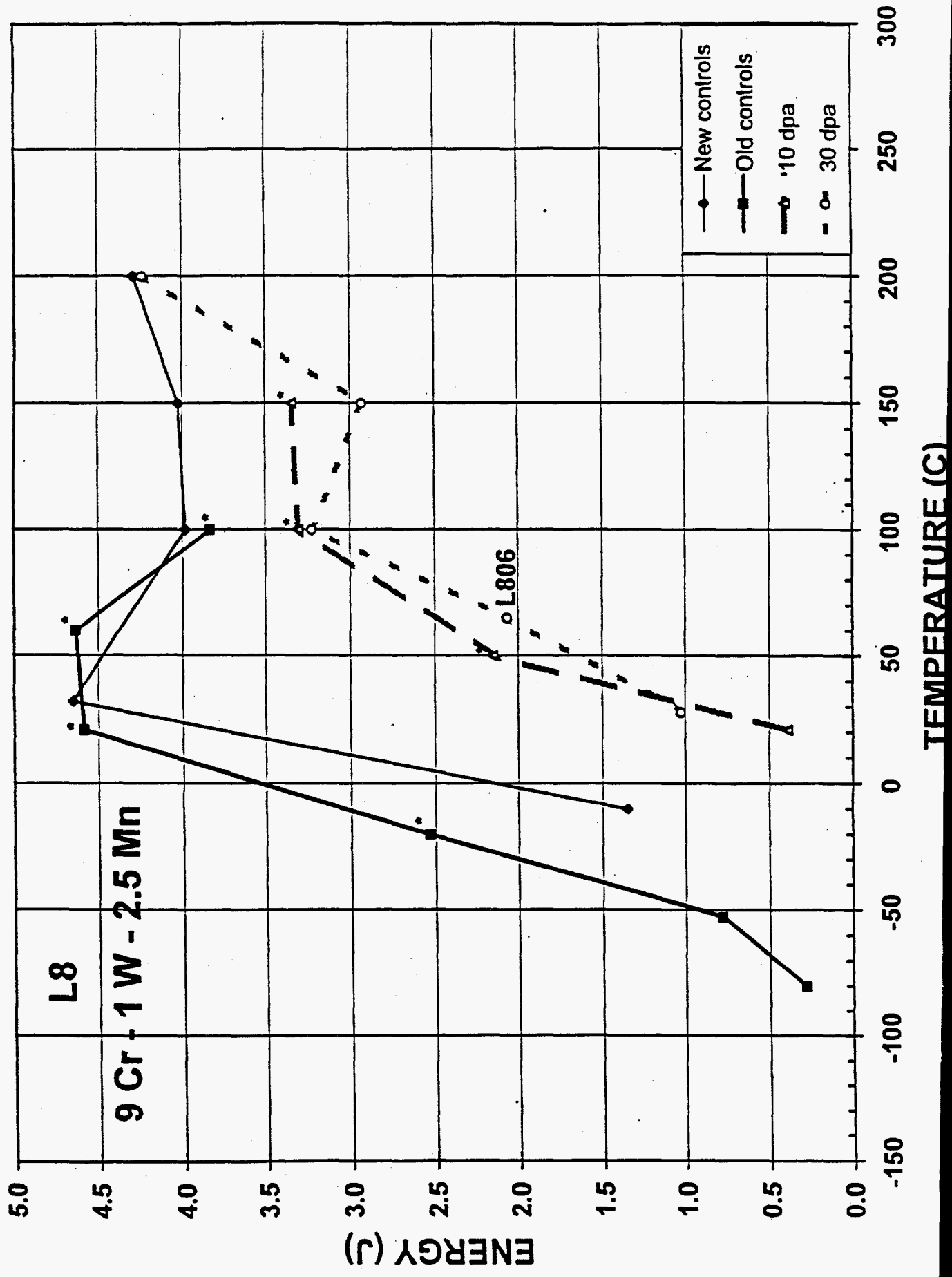
- Tests performed in an instrumented drop tower
- In flowing argon (heating) or nitrogen (cooling)
- Striker velocity  $\sim 2$  m/sec
- Span = 18 mm
- $\sim 2$  seconds between time of specimen exit from temperature control chamber to impact
- Load cell data acquired for 8 msec using a digital oscilloscope with resolution of  $2 \mu\text{sec}$
- Data transferred to desktop computer, converted to load vs. displacement, and integrated to obtain energy of impact

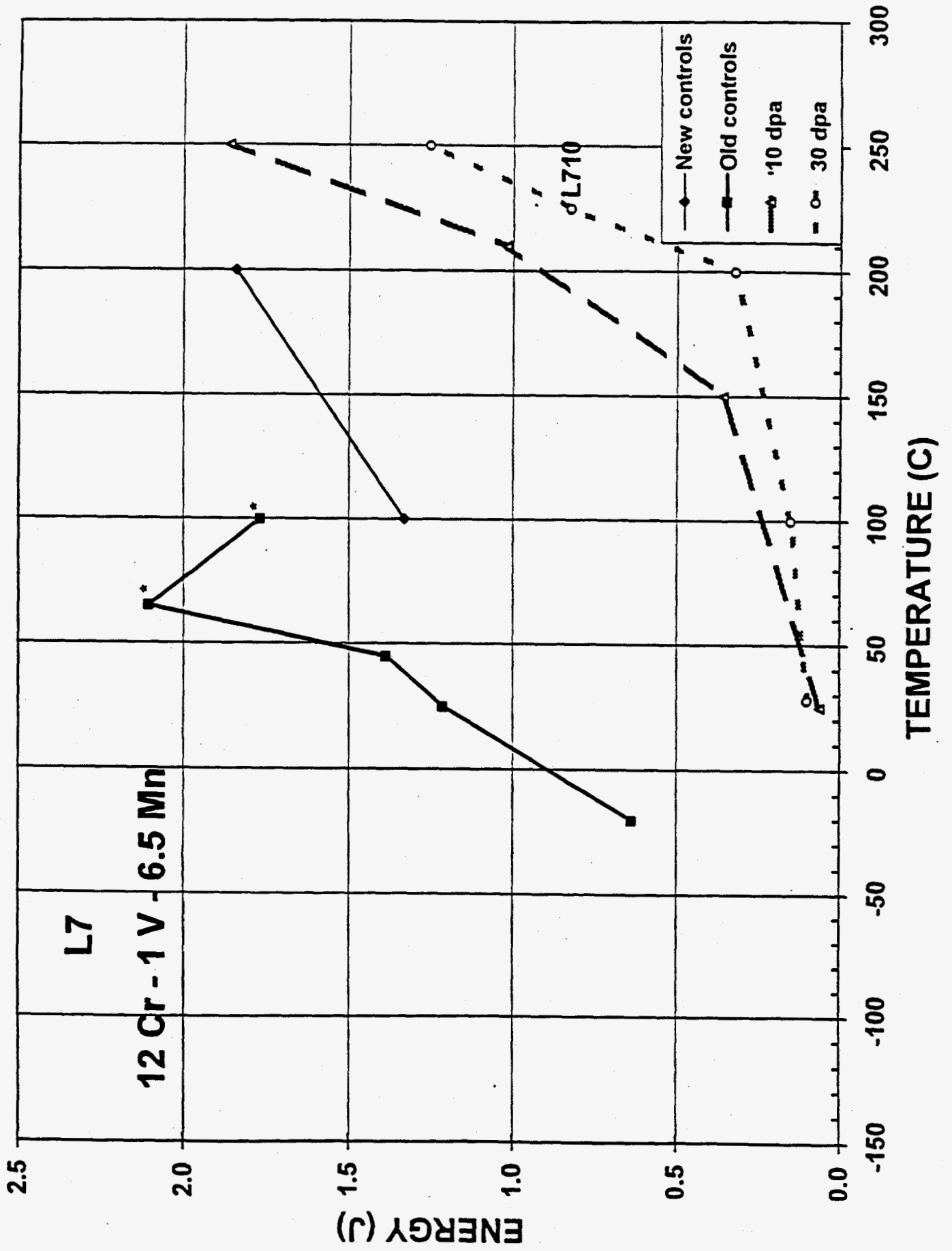


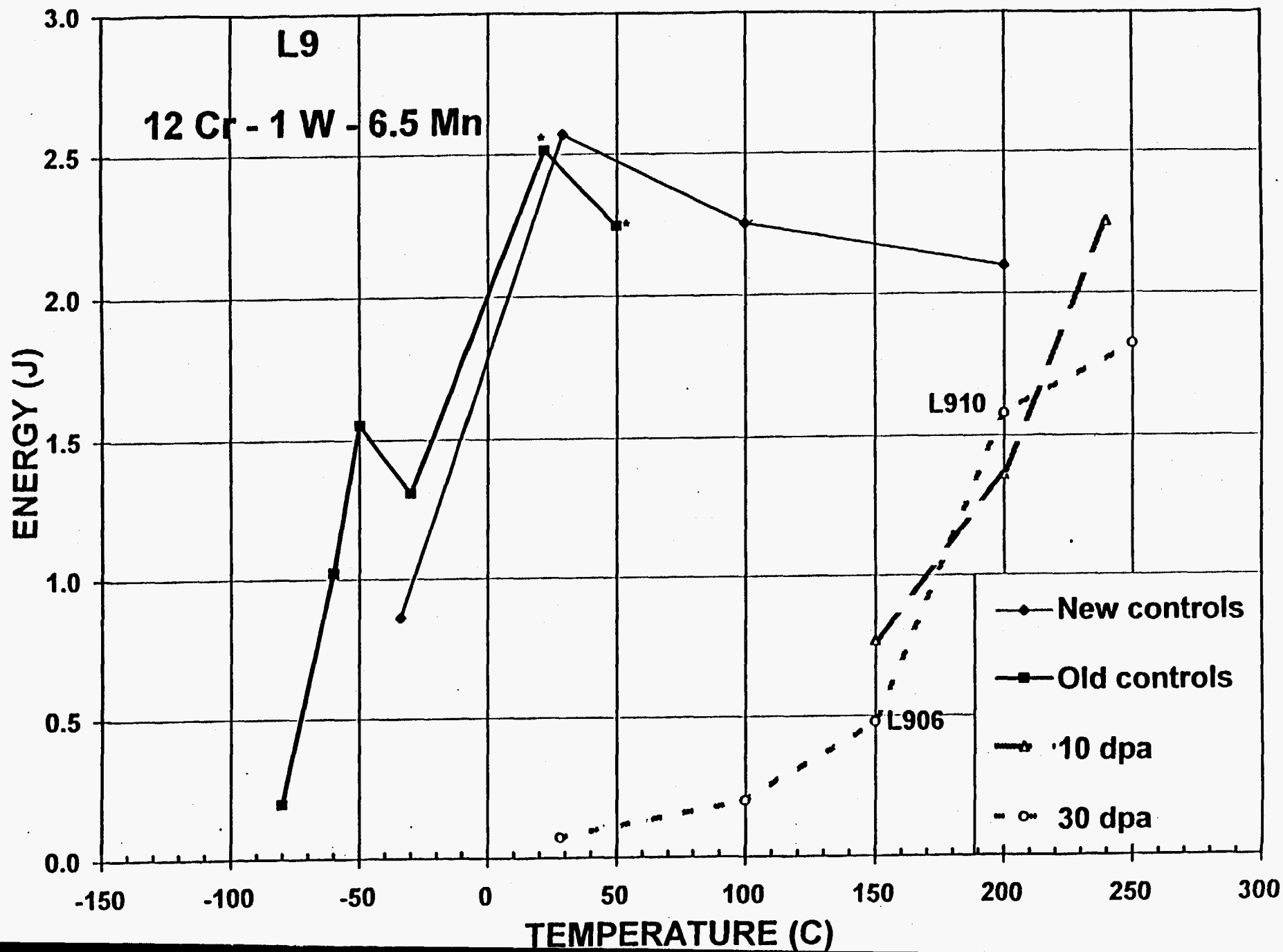






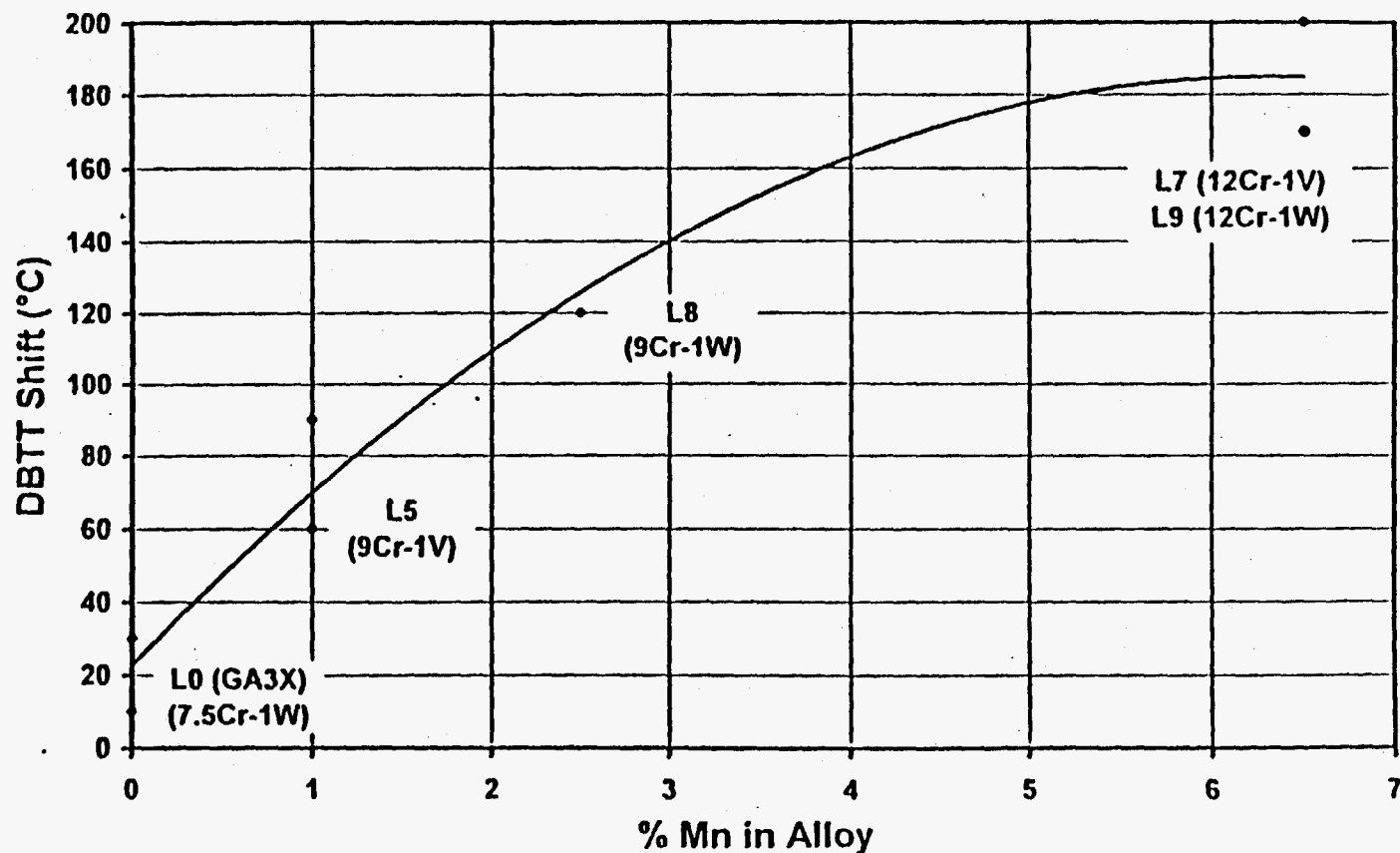






## Effect of Mn

- Mn LEVELS IN FERRITIC ALLOYS WILL INCREASE DUE TO TRANSMUTATION ABOUT 1% PER 200 DPA



IRRADIATION AT 370°C TO 10-30 DPA IN FFTF FOR 7-12Cr ALLOYS



# CONCLUSIONS

- DEGRADATION OF IMPACT PROPERTIES IN THIS ALLOY CLASS SATURATES BY 10 dpa AT  $\sim 375^{\circ}\text{C}$
- MANGANESE ADDITIONS CHANGE FRACTURE MODE FROM CLEAVAGE TO INTERGRANULAR
- PROMISE OF 7-9Cr ALLOY CLASS IS MAINTAINED UP TO 30 dpa



## **Phase II: IMPACT BEHAVIOR OF F82H AND RELATED ALLOYS FOLLOWING IRRADIATION in FFTF MOTA**

**Before the shutdown of FFTF, it was possible to arrange to irradiate an early heat of F82H, along side an improved heat of GA3X, HT-9 and a 12Cr duplex alloy called GA4X**

### **EXPERIMENTAL DETAILS**

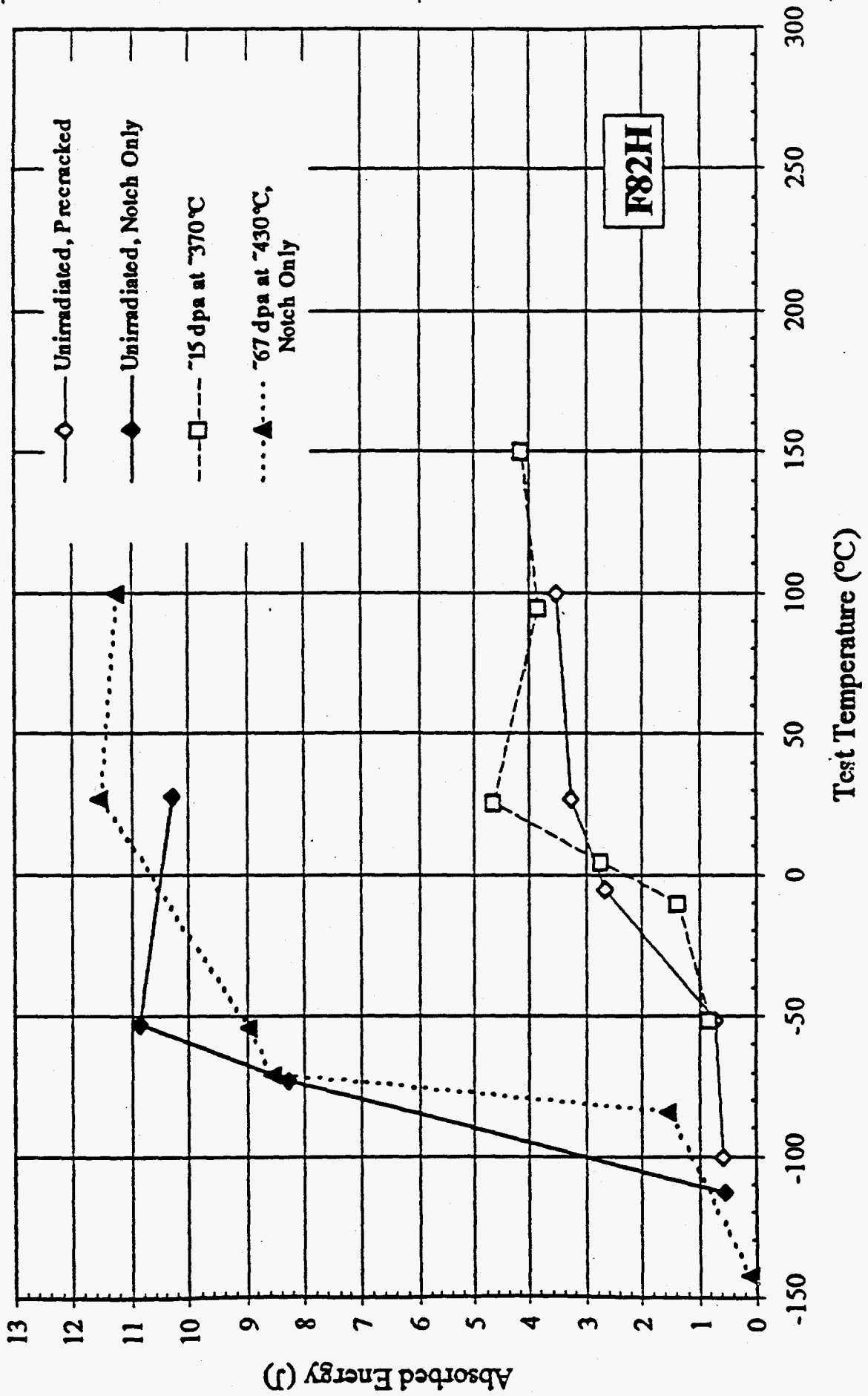
<b>ALLOYS:</b>	<b>F82H</b>	<b>7.9Cr-2W</b>
	<b>GA3X</b>	<b>9Cr-2W</b>
	<b>HT-9</b>	<b>12Cr-1Mo-0.5W</b>
	<b>GA4X</b>	<b>11Cr-2W*</b>

**heat treatments: 1000°C/20hr + 1100°C/10min + 700°C/2hr  
\*1000°C/20hr + 975°C/10min + 700°C/2hr**

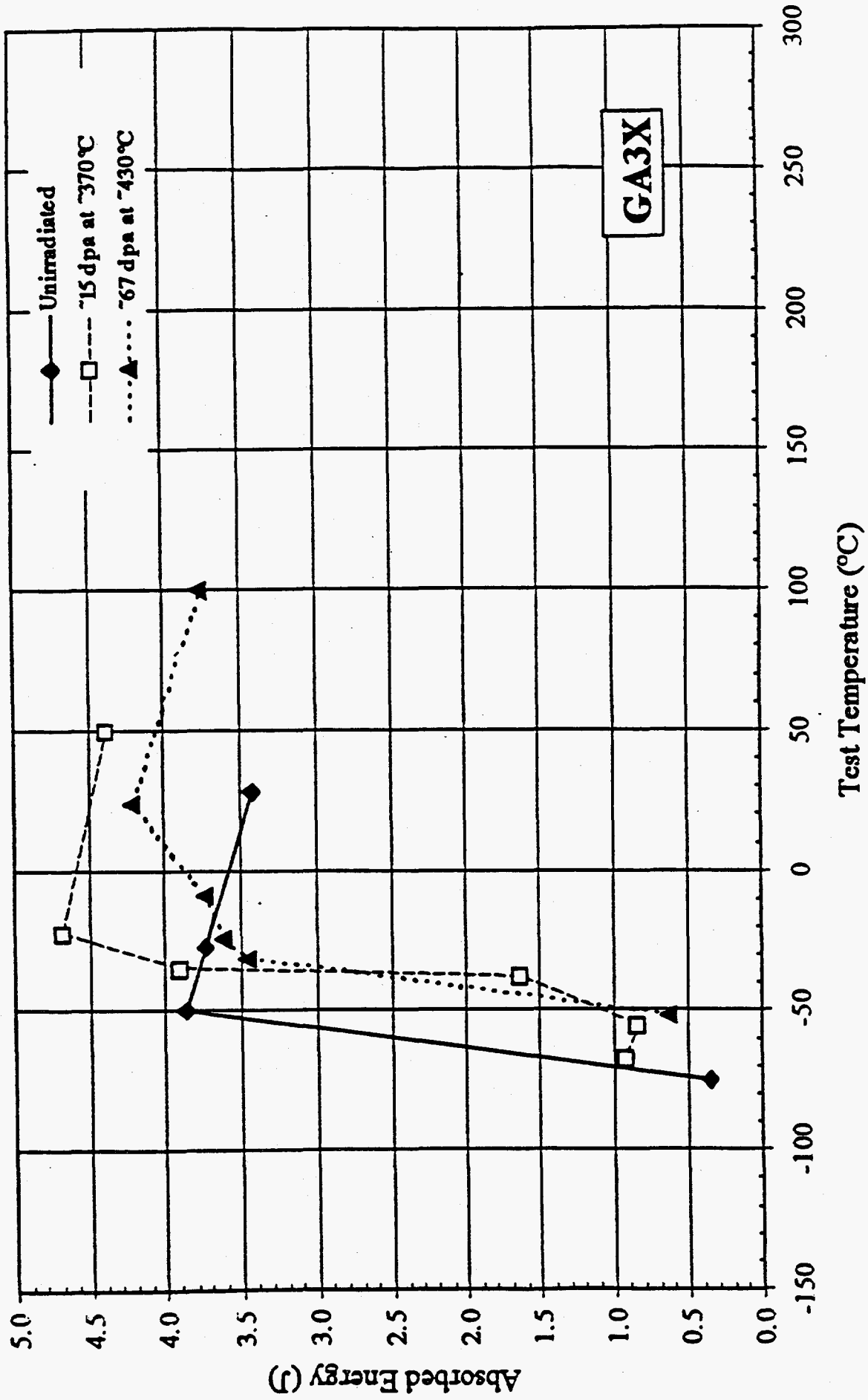
### **IRRADIATION CONDITIONS**

**15 DPA AT 370  
67 DPA AT 430°C**

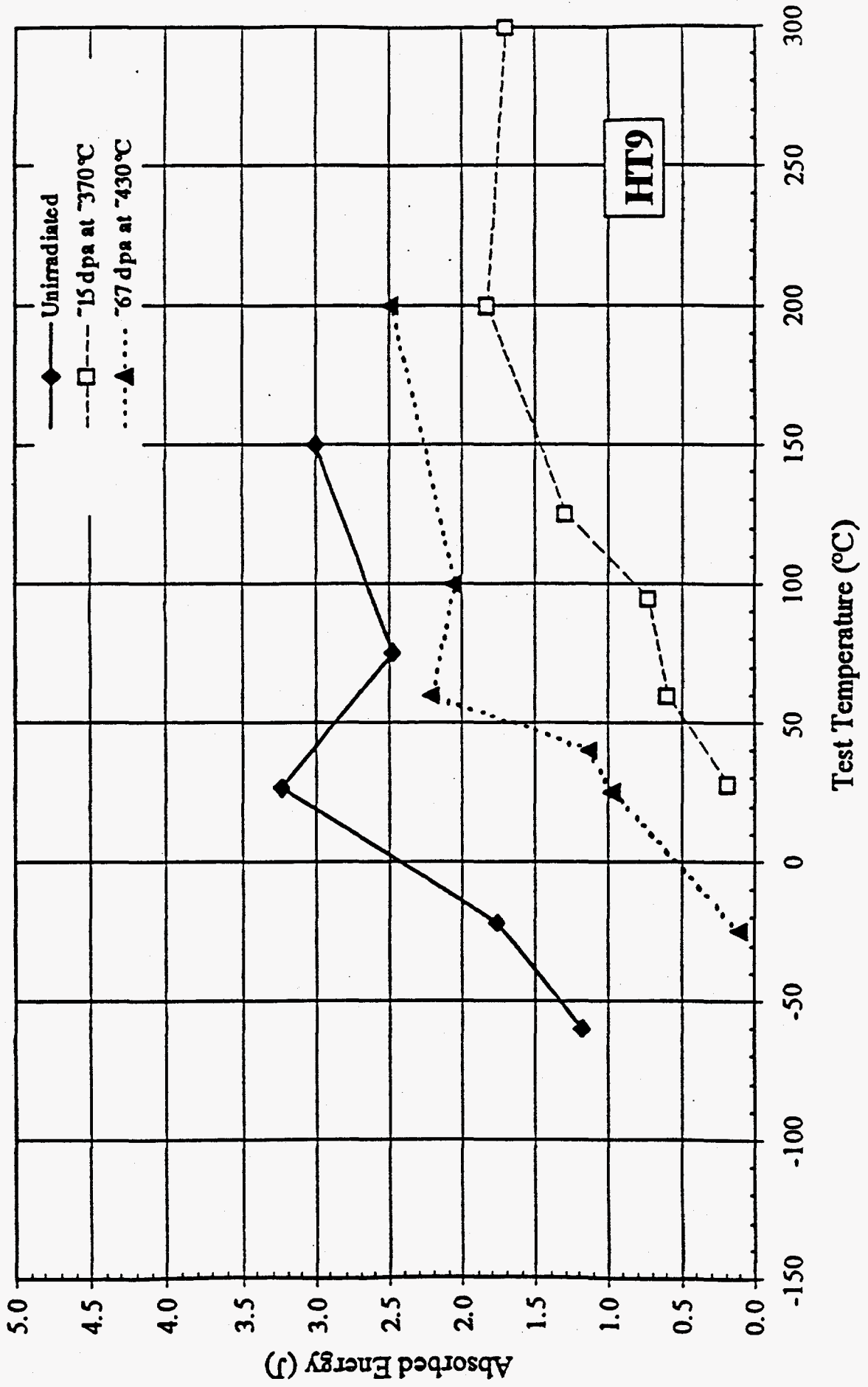
# F82H



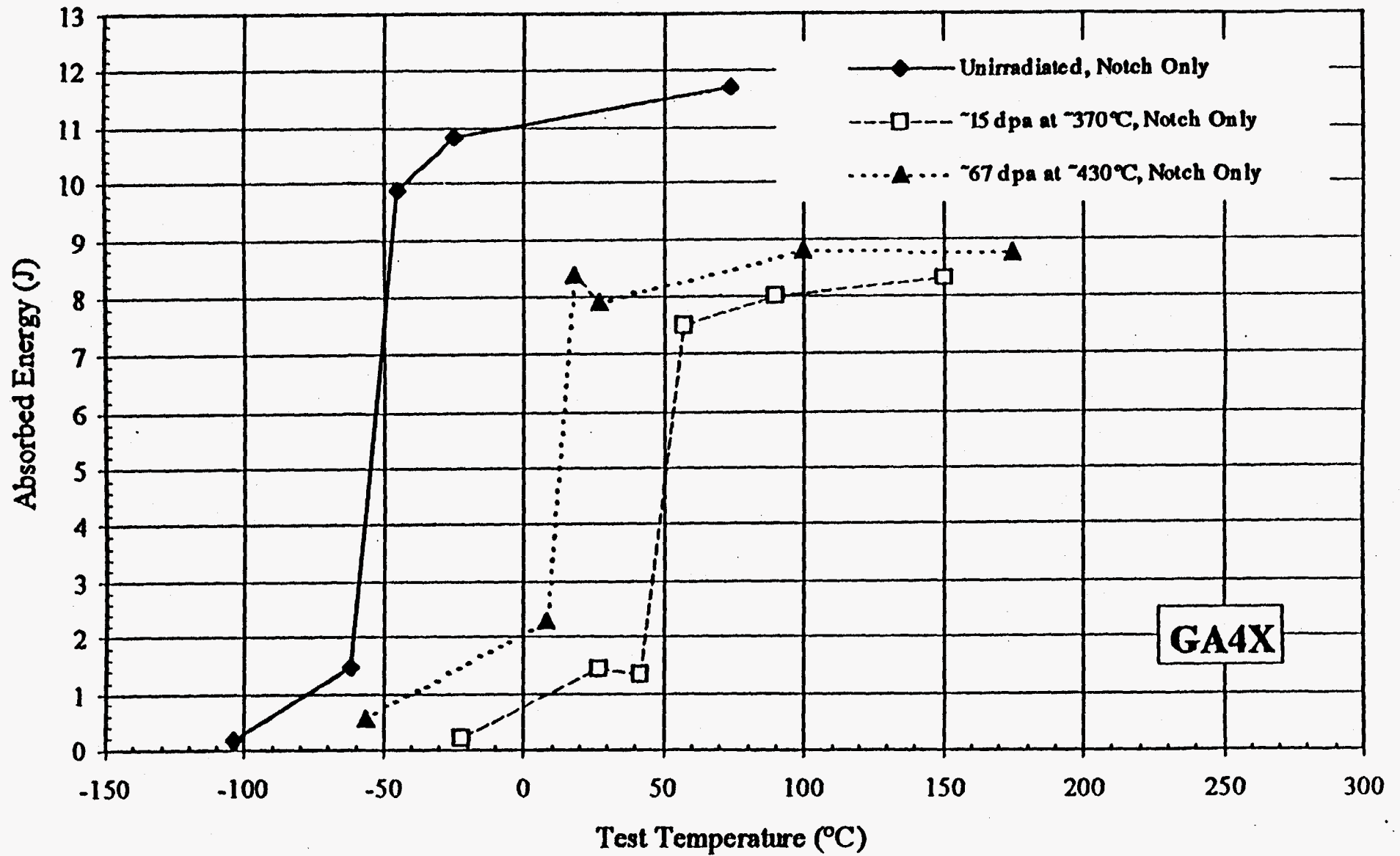
# GA3X



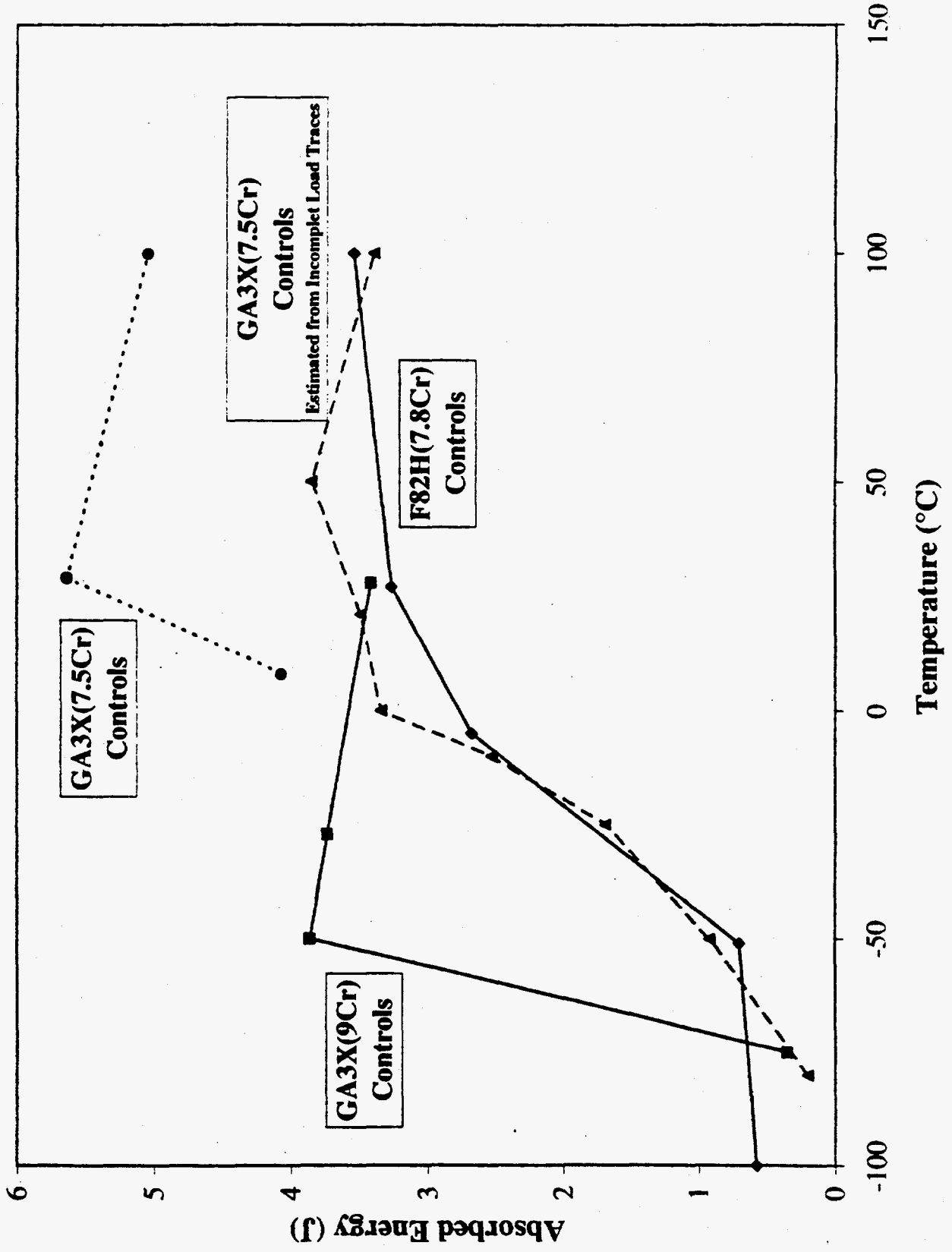
# HT-9



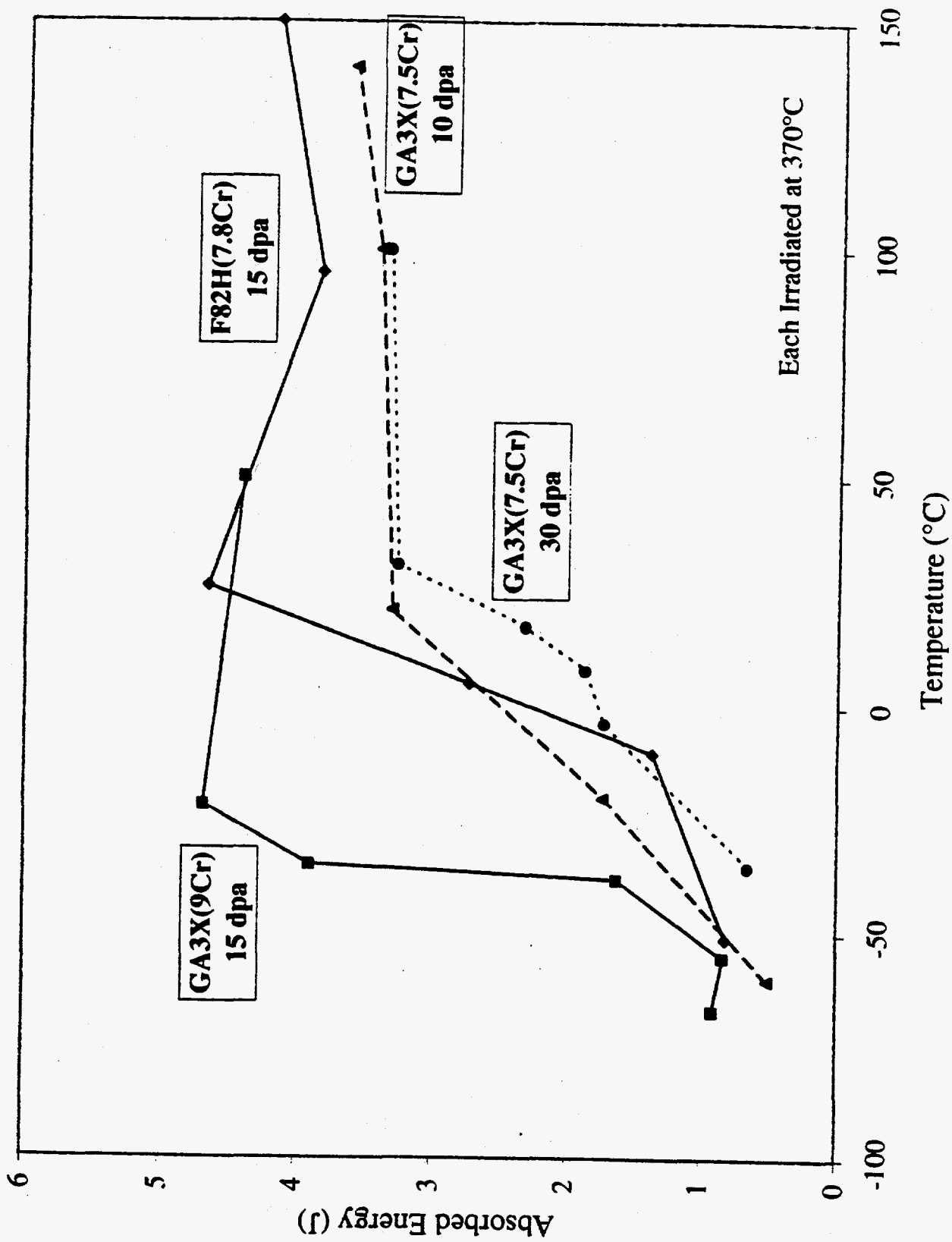
# GA4X



# Effect of Chromium Content on Charpy Response



# Effect of Chromium Content on Charpy Response



TENSILE AND CHARPY PROPERTIES OF  
IRRADIATED REDUCED-ACTIVATION  
FERRITIC STEELS

---

**R. L. Klueh**

Oak Ridge National Laboratory

Working Group Meeting on  
Ferritic/Martensitic Steels

24-25 October 1996



# OBJECTIVE OF WORK ON REDUCED- ACTIVATION FERRITIC STEELS

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- Determine the effect of composition on properties of reduced-activation Cr-W steels.
- Examine the effect of irradiation on properties of the steels.
- Optimize composition for strength, toughness, and irradiation resistance.

# REDUCED-ACTIVATION STEELS HAVE BEEN IRRADIATED TO 26-29 DPA

---

- Eight steels with 2.25-12% Cr were produced.
- Miniature tensile and Charpy specimens were aged at 365°C to 5000, 10000, and 20000 h.
- Tensile specimens were irradiated in FFTF to 15-17 and 26-29 dpa at 365°C; previous irradiations were to 6-8 dpa. Tensile tests were at 365°C.
- Charpy specimens were irradiated in FFTF to 26-29 dpa at 365°C in FFTF; previous irradiations were to 6-8, 13-15, and 20-24 dpa.

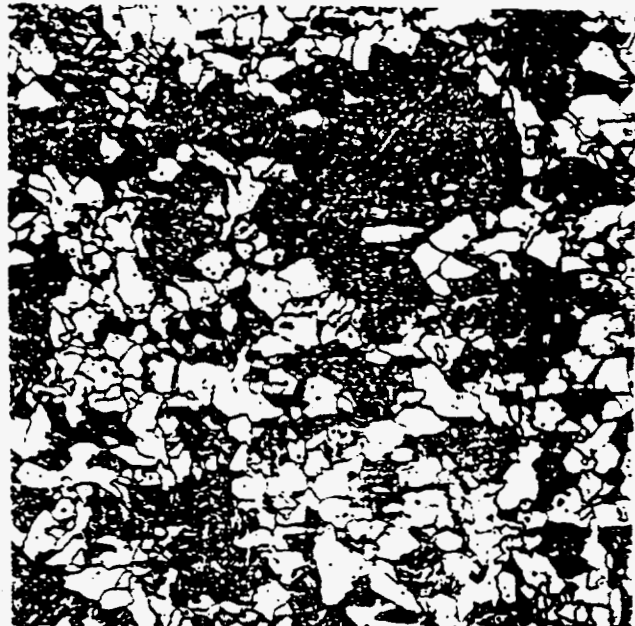
# ORNL LOW-CHROMIUM STEELS INVESTIGATED

<u>ALLOY</u>	<u>CHEMICAL COMPOSITION (WT %)</u>				
	<u>Cr</u>	<u>V</u>	<u>W</u>	<u>Ta</u>	<u>C</u>
2 1/4CrV	2.25	0.25			0.1
2 1/4Cr-2W	2.25		2		0.1
2 1/4Cr-1WV	2.25	0.25	1		0.1
2 1/4Cr-2WV	2.25	0.25	2		0.1

# MICROSTRUCTURES OF ORNL LOW-CHROMIUM Cr-W-V STEELS



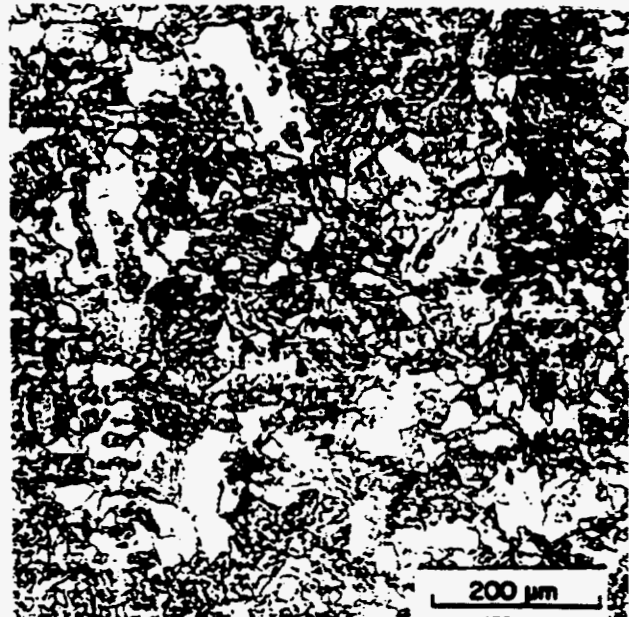
2 1/4 CrV



2 1/4-1 WV



2 1/4 Cr-2 W



2 1/4 Cr-2 WV

- NORMALIZED MICROSTRUCTURES OF 2 1/4 CrV, 2 1/4 Cr-1 WV, AND 2 1/4 Cr-2 WV STEELS CONTAINED 30, 35, AND 8% FERRITE, RESPECTIVELY, BALANCE BAINITE. 2 1/4 Cr-2 W WAS 100% BAINITE

# ORNL HIGH-CHROMIUM STEELS INVESTIGATED

<u>ALLOY</u>	<u>CHEMICAL COMPOSITION (WT %)</u>				
	<u>Cr</u>	<u>V</u>	<u>W</u>	<u>Ta</u>	<u>C</u>
5Cr-2WV	5.0	0.25	2		0.1
9Cr-2WV	9.0	0.25	2		0.1
9Cr-2WVTa	9.0	0.25	2	0.07	0.1
12Cr-2WV	12.0	0.25	2		0.1

# MICROSTRUCTURES OF ORNL HIGH-CHROMIUM Cr-W-V STEELS



5 Cr-2 WV



9 Cr-2 WV



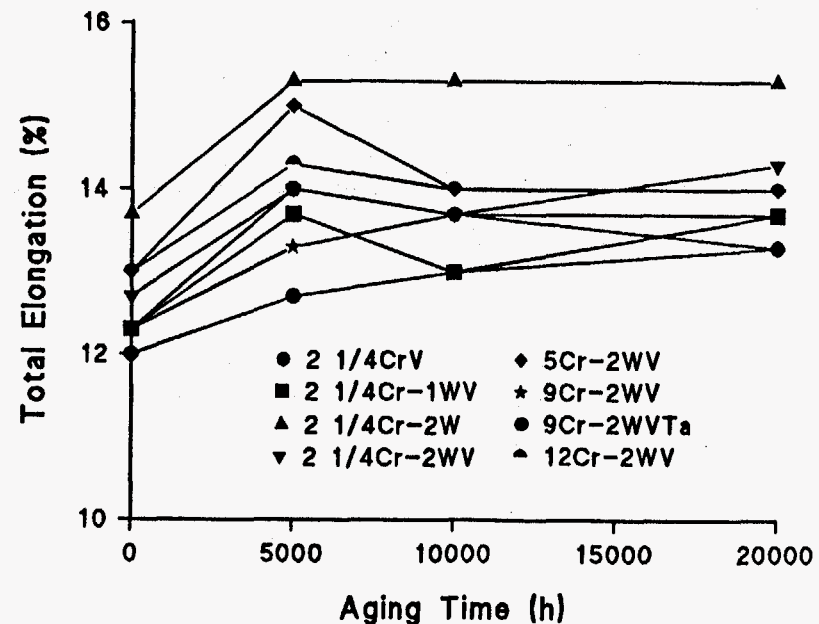
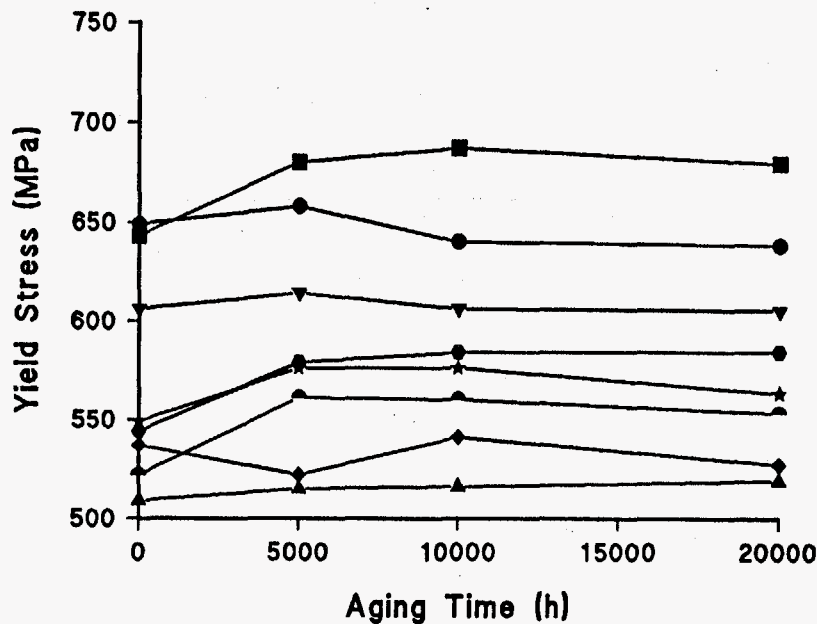
9 Cr-2 WVTa



12 Cr-2 WV

- NORMALIZED MICROSTRUCTURES OF 5 Cr-2 WV, 9 Cr-2 WV, AND 9 Cr-2 WVTa ARE 100% MARTENSITE
- NORMAL ZED 12 Cr-2 WV CONTAINS 25%  $\delta$ -FERRITE, BALANCE MARTENSITE

# THERMAL AGING AT 365°C HAD LITTLE EFFECT ON TENSILE PROPERTIES



- UTS behaved like the yield stress.
- Ductility reflected the strength properties

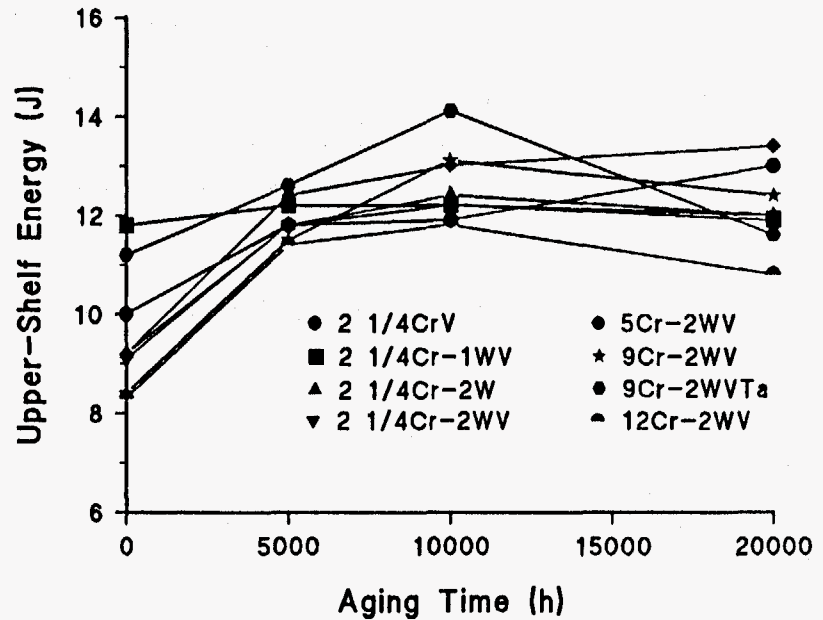
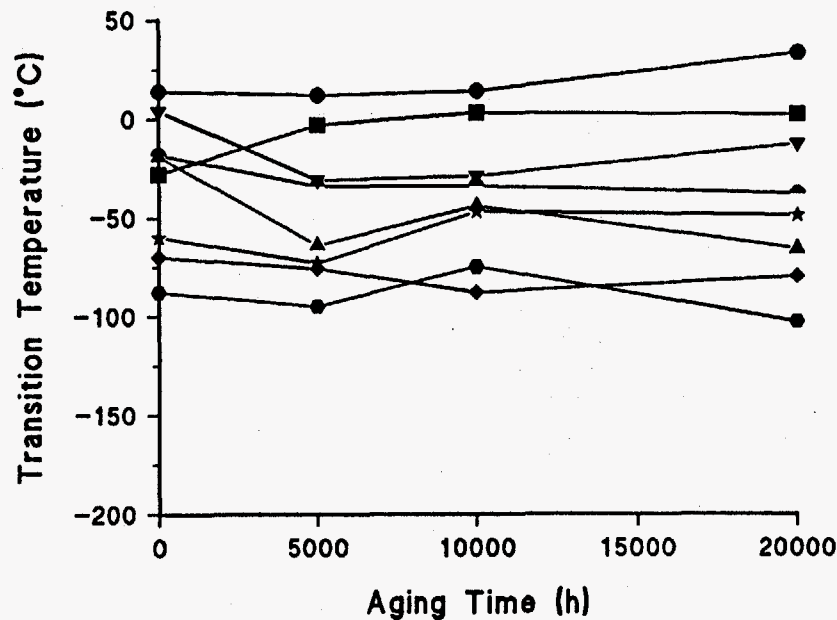
# THERMAL AGING AT 365°C HAD LITTLE EFFECT ON TENSILE PROPERTIES

---

- 2 1/4Cr-2W was weakest because it contained no vanadium for carbide dispersion strengthening.
- Vanadium-containing low-Cr steels (2 1/4CrV and 2 1/4Cr-1WV) tempered at 700°C were stronger than steels tempered at 750°C.
- The 2 1/4Cr-2WV steel was the strongest of the steels tempered at 750°C (5Cr-2WV, 9Cr-2WV, 9Cr-2WVTa, and 12Cr-2WV).

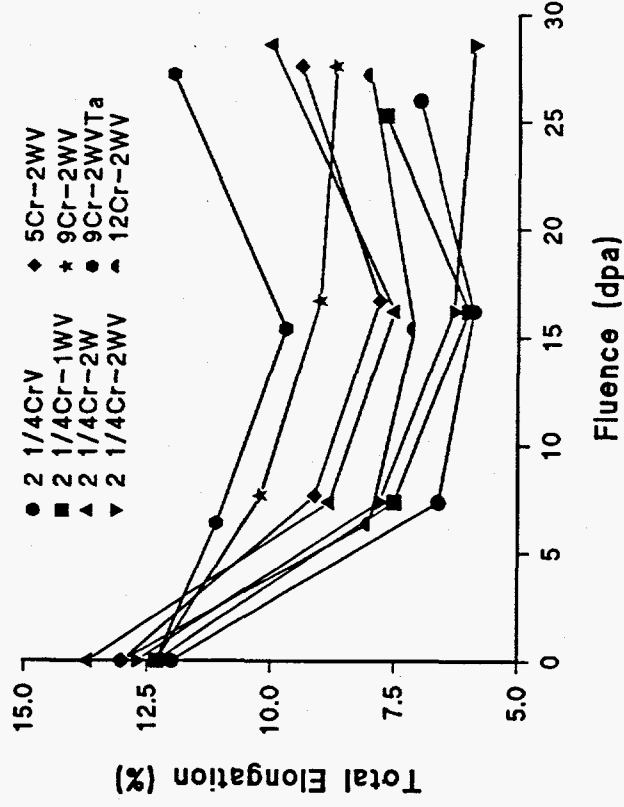
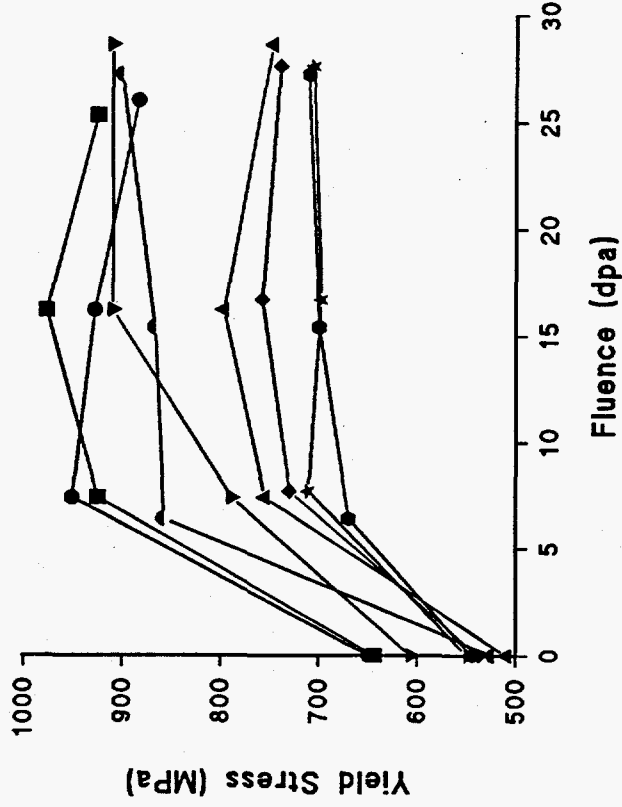


# THERMAL AGING AT 365°C HAD LITTLE EFFECT ON CHARPY PROPERTIES



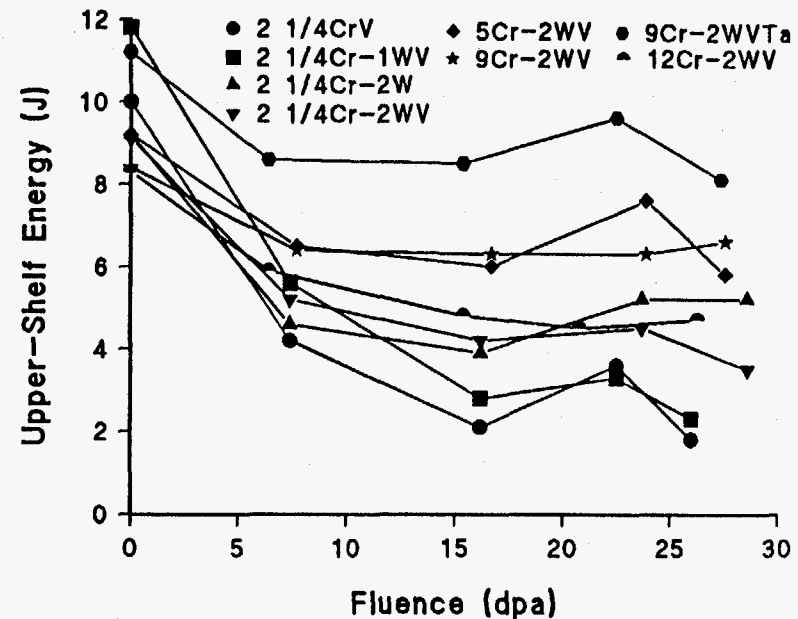
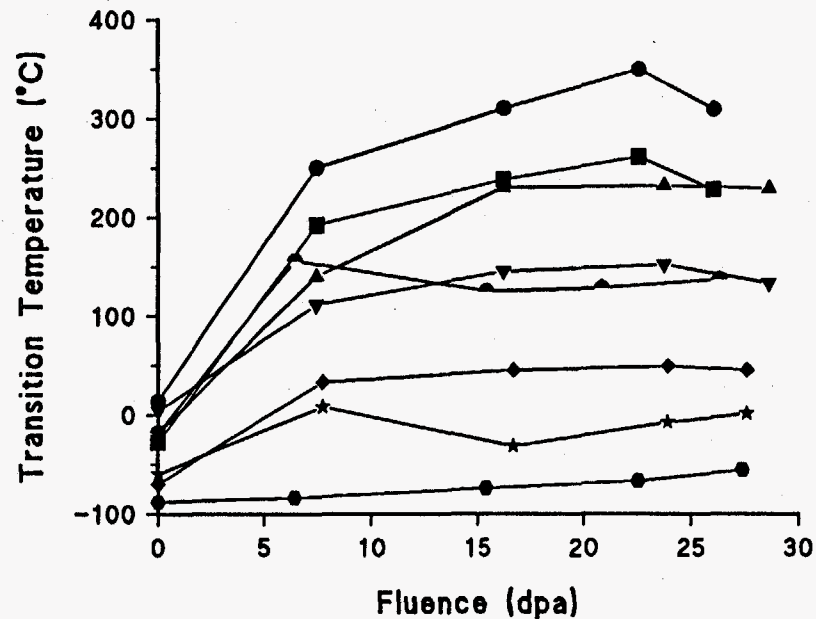
- Upper-shelf energy of several steels showed a slight increase, usually in initial 5000 h.

# IRRADIATION HARDENING SATURATES WITH FLUENCE



- Hardening is accompanied by a loss of ductility.
- Low-chromium and 12Cr-2WV steels hardened most.

# HARDENING CAUSED A CHANGE IN CHARPY PROPERTIES



- Duplex microstructures showed largest changes.
- The 9Cr-2WVTa has the best properties, but the transition temperature increases with fluence.

# DUPLEX MICROSTRUCTURE CAUSES INFERIOR PROPERTIES

---

- Higher unirradiated DBTT of 2.25Cr steels and 12Cr-2WV is caused by the duplex microstructure.
- Anderko et al. showed that carbides on ferrite/martensite interfaces adversely affect toughness.
- The 12Cr-2WV and 2.25Cr steels contain precipitates along ferrite/martensite and ferrite/bainite interfaces, respectively.

# LOW-CR STEELS CAN BE IMPROVED BY CHANGE IN MICROSTRUCTURE

---

- Ferrite can be eliminated from the bainitic steels by heat treatment and alloying.
- Previous work showed the 2 1/4Cr-2WV steel had improved properties in unirradiated condition after heat treating to develop a bainitic microstructure.
- Low-Cr steels have been developed that have improved impact toughness (improvement over 9Cr-2WVTa).

## 9Cr-2WVTa STEEL SHOWS BEST PROPERTIES FOR STEEL OF THIS TYPE

---

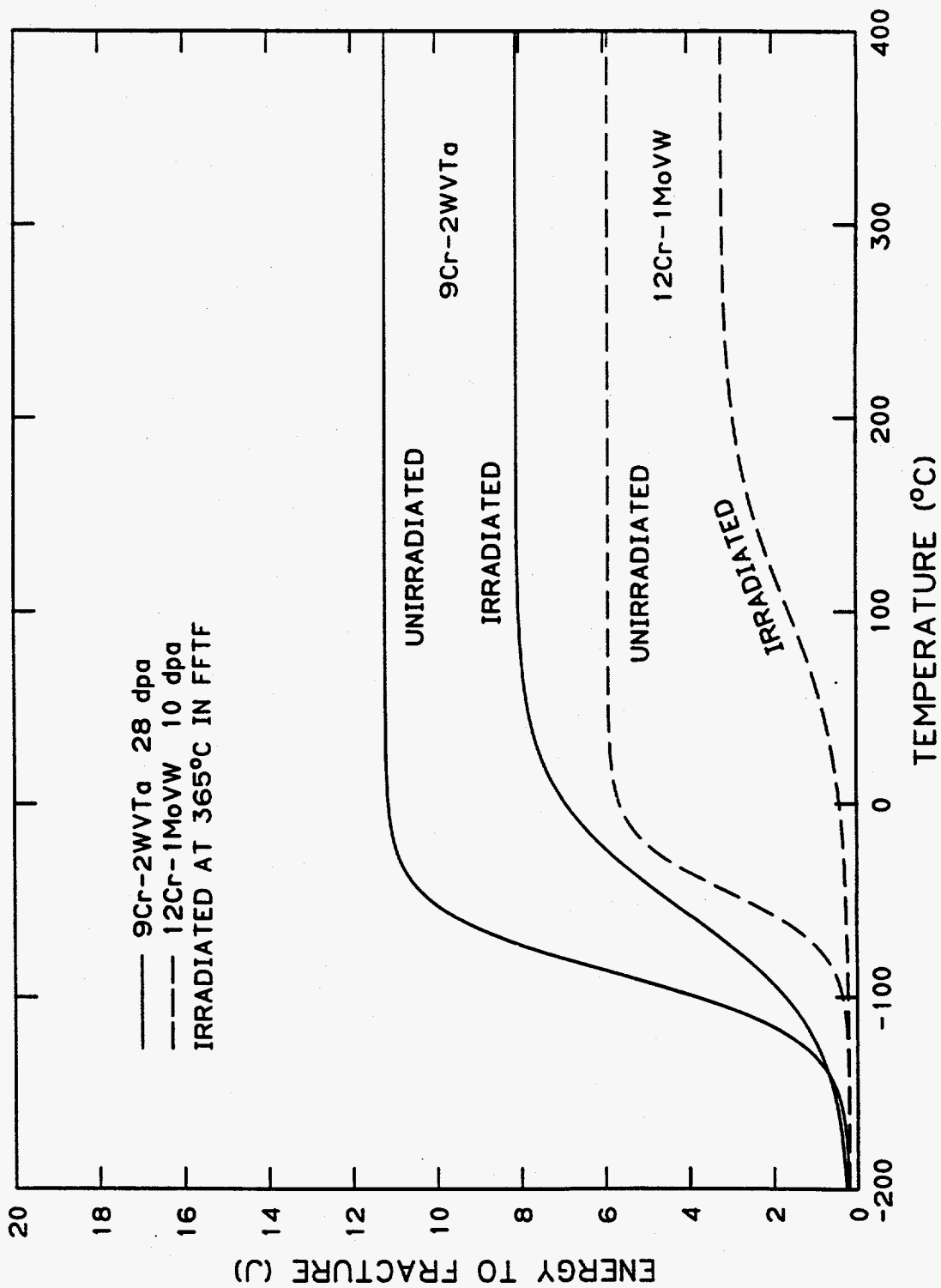
- 9Cr-2WVTa shows small shift in DBTT (32°C) and has low unirradiated DBTT.
- TEM indicates little difference in microstructure of normalized-and-tempered 9Cr-2WV and 9Cr-2WVTa prior to irradiation.
- Tantalum refines prior-austenite grain size, but lath size of the two steels was similar.
- Precipitate in Ta-containing steel did not affect the strength, which was same as steel without Ta.

## TANTALUM LEADS TO IMPROVED PROPERTIES OF 9Cr-2WVTa

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- TEM and atom probe indicates that most of the Ta remains in solution in 9Cr-2WVTa.
- Difference in Charpy properties of 9Cr-2WV and 9Cr-2WVTa before and after irradiation occurs despite same strength and microstructure.
- Irradiation produced similar changes in both steels.
- Excellent properties of 9Cr-2WVTa attributed to Ta in solution.
- Increase in DBTT with fluence would follow if Ta is removed from solution during irradiation.

# 9CR-2WVTa IS IMPROVEMENT OVER CONVENTIONAL STEELS





## SUMMARY AND CONCLUSIONS

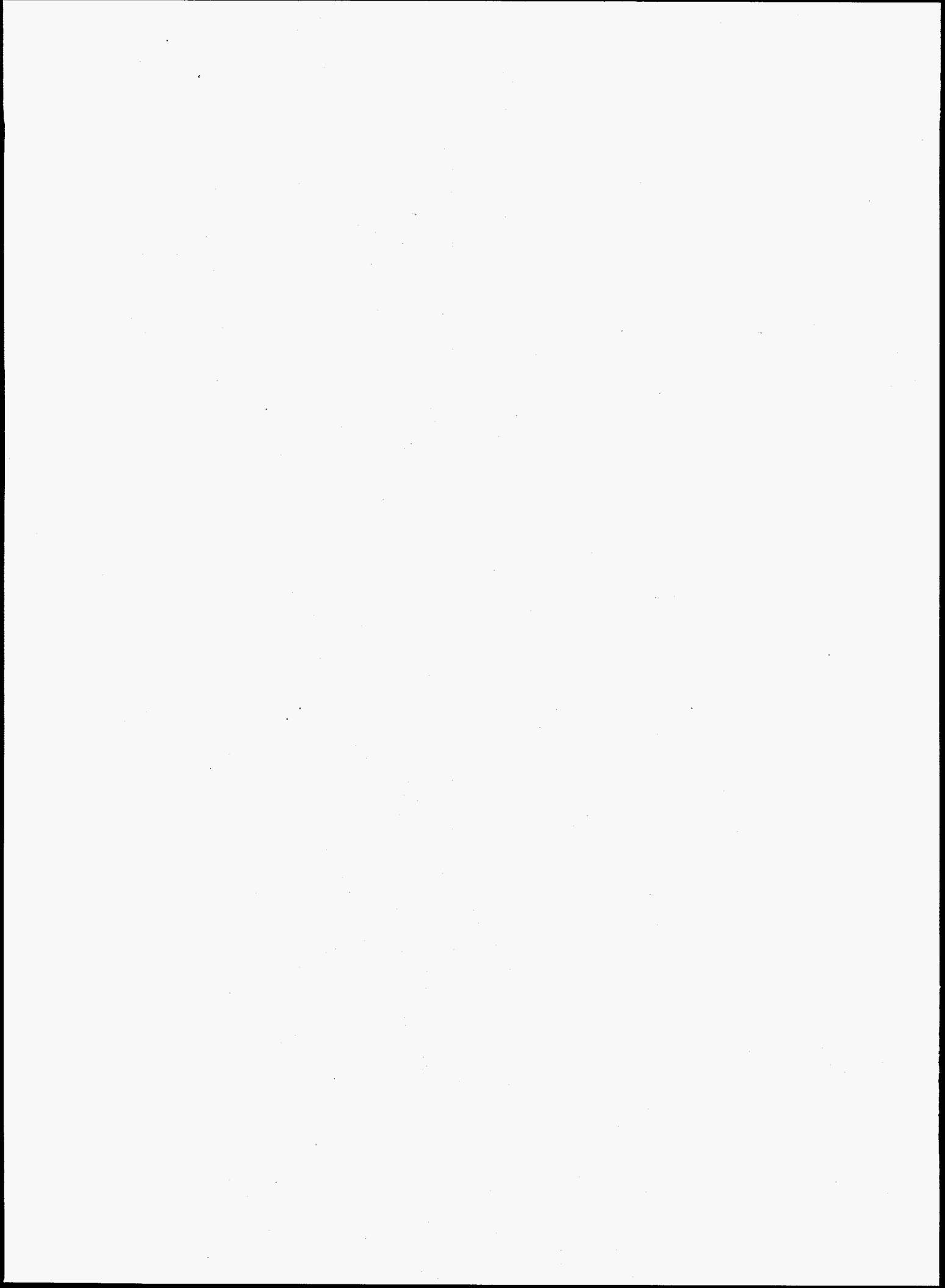
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- Tensile and Charpy specimens of Cr-W steels with 2.25, 5, 9, and 12% Cr were irradiated to 29 dpa at 365°C in FFTF.
- Irradiation hardened all steels, which produced an increase in DBTT.
- The DBTT shift was affected by the microstructure, with duplex microstructures of the 2 1/4Cr steels and the 12Cr-2WV steel showing the largest shifts.

## SUMMARY AND CONCLUSIONS

---

- Alloy development to produce steels with 100% bainite should improve irradiation resistance of low-Cr steels.
- The 9Cr-2WVTa steel had a shift of 32°C (the smallest shift ever observed for this type of steel under these irradiation conditions), compared to a 56°C shift for 9Cr-2WV, the second lowest shift.
- The superior behavior of 9Cr-2WVTa was attributed to Ta in solution.



## Re-orientation of the EU Material Programme in 1996 for the Period 97/98

W. Dietz\*, EU Commission, DG 12, Fusion Directorate

The long term material program within the European Fusion Technology Program was reviewed in 1996 on request by the Fusion Technology Steering Committee (FTSC) and programmatically due to Program Milestone NR. 2. In this paper the status of this review is presented. Final approval of the draft proposal of the revised material program by the FTSC will be in November 96.

The material program was established in 1994 by the Task Force Material (nominated representatives of European laboratories for the Fusion Program). Details of the material program have been presented during the IEA Working Group meeting in Feb. 1995/ORNL, US. The selection of a primary candidate alloy from a group of reduced activation (RA) ferritic/martensitic(F/M) steels was defined as the main goal. In parallel some limited scoping activities on other potential low activation materials such as a Vanadium alloy V-4Cr-4Ti and the ceramic composite SiC/SiC are part of the program to allow an evaluation for the constraints on the use of these materials in a DEMO blanket. The overall budget for the period 95/98 is about 29 MECU.

In the EU Fusion Technology Program up to 1995 four different DEMO blanket concepts have been considered. During the Blanket Selection Exercise in 95 the four concepts were reduced to two options, the Water Cooled Lithium Lead (WCLL) concept and the He Cooled Pebble Bed (HCPB) concept. It was decided by the Fusion Technology Steering Committee (FTSC-P) that after the blanket selection exercise the blanket and the material program should merge in a European Blanket Project (EBP). The main objective of the EBP will be the construction of the DEMO relevant blanket test objects in ITER. A survey about the common material and blanket program was recently presented during the SOFT-19 conference in Sept.96 in Lisbon by Giancarli et al.( a copy of the invited paper was distributed to the participants of this WG meeting at Culham/JET facilities).

The re-orientation of the long-term material program means that the previously elaborated and approved program in 94/95 was reviewed and separated in two elements with a different schedule and different technical boundary conditions . i) The program related to the EBP with the near-term ITER Test Modules (ITM) characterized by environmental condition of 3-15 dpa (dose levels not finally detailed for the Extended Performance Phase / EPP), operation temperatures in the range of 250-500°C, pressurized water or He as coolant and ii) the Advanced Material Program (AMP) related to long term requirements of a Fusion Reactor (characterized by high neutron dose e.g. 100 dpa or even much more and high temperatures of about 550°C).

All tasks on advanced metallic and ceramic LA material (V, Ti,SiC...) have been grouped in the longer term AMP (working package WP2 and WP3 in the 95 approved program). Tasks on ferritic/martensitic steel related to DEMO (e.g. high dose irradiation in Phenix, or basic investigations with neutron scattering on microstructure) will however be grouped under the EBP project in a specific working package (WP 6) for DEMO relevant tasks. In the attached chart (draft) a survey is presented for the common material and blanket development program for the EBP (in this chart ferritic/martensitic activities with longer term aspects have been included also in the long term advanced material program with regard to high dose IFMIF irradiation).

\*)Work performed under Contract NET/95-886 of the European Commission to IKL, D-51503 Roesrath / FRG

A survey about the new working packages of the material program is attached too.

The new work break down structure was oriented on EBP project related tasks and the needs to verify the materials performance for the EBP operating conditions and fabrication of components of the ITMs. The selection of a primary candidate alloy for end of 98 was defined as a main goal in the already approved program for 95/98. This objective will be also a milestone for the qualification of the F/M steel for ITMs. All ongoing activities for this objective can be fully integrated in the new EBP program with its working packages, tasks and subtasks (contributions of laboratories to the tasks). Re-orientation of the program means therefore that we have three categories of activities: I) Activities which are not changed (the bulk), II) Activities with adaptations (minor modification due to new technical conditions or postponement due to lower priorities), III) Activities which are new. In the attached chart the additional subtasks have been indexed (see foot note in the table). In total the changes in the program are about 15 % of the EBP related budget.

In the following the Working packages in the new EBP material program are briefly described and commented.

All the subtasks and activities of EU laboratories related to the selection of the primary candidate alloy (PCA) will only be re-ordered without a change technically as mentioned. This selection process for a PCA will follow the guidelines of the previous planning from 1994/95 and will be based mainly on the results of the new working packages

#### **WP1 Irradiation experiments for RA\_F/M steels and**

#### **WP2 Metallurgical and mechanical and characterization of RA\_F/M**

It was anticipated from previous experiences that variations in alloy chemistry (e.g. content of Ta or W, Ta or Ti ...) may have a significant impact on the mechanical behavior, also the thermo-mechanical treatment respectively the fabrication process. There is presently no reason in reducing the number of the F/M options in the program.

Neutron fluence in the experiments in WP 1 will go in 1998 up to 2.4.dpa and will cover the range of ITM in the first operational phase. The alloy selection will be based mainly on results from tests on the low temperature toughness measured by impact tests in the range of 200 - 450 °C after irradiation.

The mechanical and metallurgical characterization of the steels in WP 2 will be based on tensile and hardness tests with some spot type testing for fatigue and creep behavior, also for comparison with the previous reference material MANET and on the mod. 9 Cr steel - the code approved F/M steel in the frame of the FBR program. Present designs are based on data mainly of MANET.

#### **Working Package 3 (WP 3): Compatibility of the F/M steels in the Fusion environment**

WP3 contains testing in water, liquid Pb-Li and for increased hydrogen concentrations in the F/M material (H-sources are e.g. corrosion and nuclear reactions,..). An increase on efforts in this technical area is due to the fact that the 8-9 CrW-Ta-V steel are a new category of steel for which the general corrosion is not known. Checking the risk of fast crack propagation (liquid metal embrittlement of the irradiated hardened structure, hydrogen cracking,..) need sophisticated approaches. For an effective use of experimental tools joint tests will be performed between the material and the WCLL sub-project. In 1994 only some generic activities were started for corrosion waiting for further definition

of a corrosion program. after the blanket selection exercise with more precise boundary conditions for tests.

Corrosion in He environment was not considered as a critical issue for the HCPB system which means that no corrosion tests presently have been planned in He atmosphere.

#### **Working package (WP4): Qualification of Fabrication**

In WP 4 additional efforts have been integrated especially for welding related tasks for the ITMs. Generic tests on weldments were already an extensive part of the existing 1995 - program (Electron Beam and Tungsten Inert Gas , TIG- welds, provided by JAERI in the frame of IEA implementing agreement). New will be the actions for filler metal development both for the F/M steel and the transition-weld between F/M steel and the austenitic steel in the WCLL. These tasks will need permanent interaction with the WCLL system engineers.

#### **Working Package (WP5) : Rules for mechanical design and design data**

This WP includes activities which were already a significant part of the planning 1994. Some adjustments have been made following the requirements of the systems which needed some additional recourses such as aspects of quality control of weldments. Material for component testing will be provided under this topic uniquely for the both sub-projects WCLL and HCPB, also to verify a preliminary specification for RA\_F/M steel. The definition of design data will need a continuous interaction with design engineers.

#### **Working Package (WP6): Qualification of RA\_F/M steel for DEMO**

In WP 6 all the longterm tasks (high dose, basic investigations,.. ) have been grouped in a separate package, considering the different needs for a DEMO and the ITMs. Due to the delay of the Phenix irradiation it was proposed to finalize the experiment with its target dose of 30 dpa in 1999. This WP includes also investigations for the understanding of deformation mechanisms (fatigue) under radiation (Ion Beam) and structural stability for high dose rates under heavy ion irradiation.

From a programmatic point of view a further review of the material activities will be made with the next Milestone (NR 3) in late 1997 with the available data after irradiation. This review will give further input for the confirmation of the direction of the program. After the selection of the PCA in 1998 material with the selected alloy composition and process requirements will be investigated in depth considering the conditions for operation and fabrication in more detail.. It is anticipated that for 2005 the selection of the RA\_F/M steel will be validated based on present assumptions for the ITER schedule.



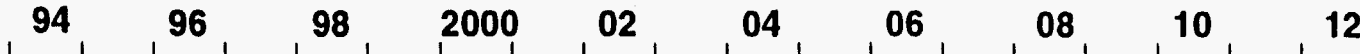
# Survey about the Structural Material Programme for the European Blanket Project

(Draft)

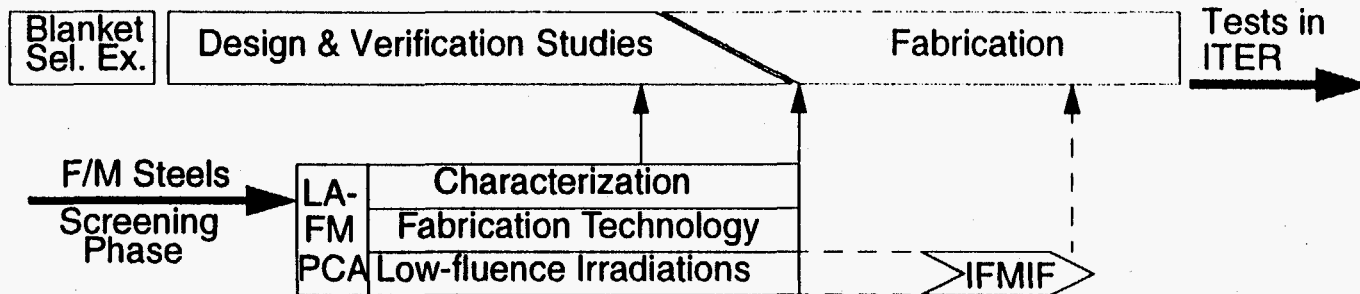
Work package	Objectives	Activities / Remarks
<b>SM1:</b> Irradiation experiments on Reduced Activation Ferr./Mart. (RAF) steels	# Selection of a primary candidate alloy based on DBTT behavior (radiation hardening) # Understanding of radiation hardening and microstructural evolution	★ Screening programme => testing sensitivity of candidate alloys (8) for low temperature embrittlement under irradiation (200-450°C); ★ Co-ordinated activity for studies on He/dpa effects on mechanical behaviour (radiation hardening) and microstructure
<b>SM2:</b> Metallurgical & mechanical characterisation of RAF steels	# Characterisation of the transformation behavior and microstructure... # Investigation of basic mechanical properties	★ Spot type tests for selected design relevant properties => Tensile, hardness, Creep, fatigue, ageing effects on toughness IEA programme on a Japanese F 82 h mod alloy
<b>SM3:</b> Compatibility of RAF steels with the fusion environment	# Description of the general corrosion behavior in water, Pb-Li and regarding hydrogen # Evaluation of the risks for corrosion cracking	★ ★ Long term Corrosion tests in Water and Pb-Li ★ ★ Cracking in water and <u>Pb-Li</u> , <u>simulating radiation hardened structure</u> ★ ★ Detailed investigation of hydrogen diffusion and embrittlement phenomena ★ Assessments on hydrogen problems and compatibility
<b>SM4:</b> Qualification of fabrication processes for RAF steels	# Qualification of processes ( base metal, EB/TIG welding, bending...) # Filler metal development	★ ★ Filler metal specification and characterisation test ★ Properties of welded joints , diffusion bonding and HIP products
<b>SM5:</b> Rules for mechanical design, fabrication and inspection	# Evaluation of life prediction models # Design data for components # Procurements specification and its verification # Fracture mechanics concept	★ Data Aquisition for design data ★ Evaluation for the application of mechanical design codes for fusion including fracture mechanics studies ★ Investigation of specimens size effects ★ ★ Efforts for procurement specification : Ordering of a RAF heat ★ ★ Evaluation of quality control of RAF weldments for ITMs
<b>SM6:</b> Qualification of RAF steels for DEMO	## Understanding of mechanisms during deformation and simultaneous irradiation # Swelling/irradiation creep	★ Irradiation in Dual beam facility (light and heavy ions) ★ High dose/high temperature irradiation in Phenix

RAF: Reduced Activation Ferritic/Martensitic steels ; \*\* ) Increased efforts 1997/1998

# Materials and Blanket Development

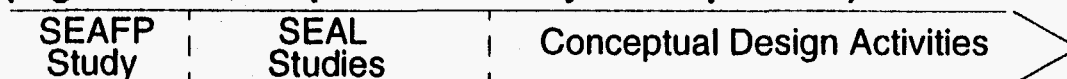


## Test Modules for ITER (Low fluence $\leq 15$ dpa; High pulse operation)

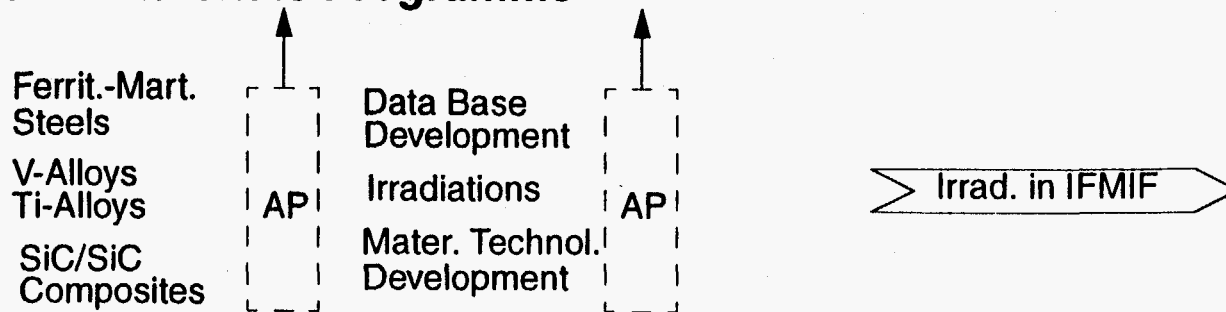


## Breeder Blankets for DEMO- and Commercial FR's

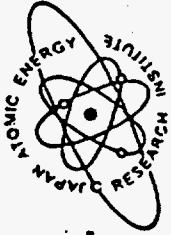
(High fluence, temperature, steady state operation)



## Longterm Materials Programme







**Present Status of JAERI Program on LAM Development**

**A. Hishinuma  
Material Science & Engineering Dep.  
JAERI, Tokai**

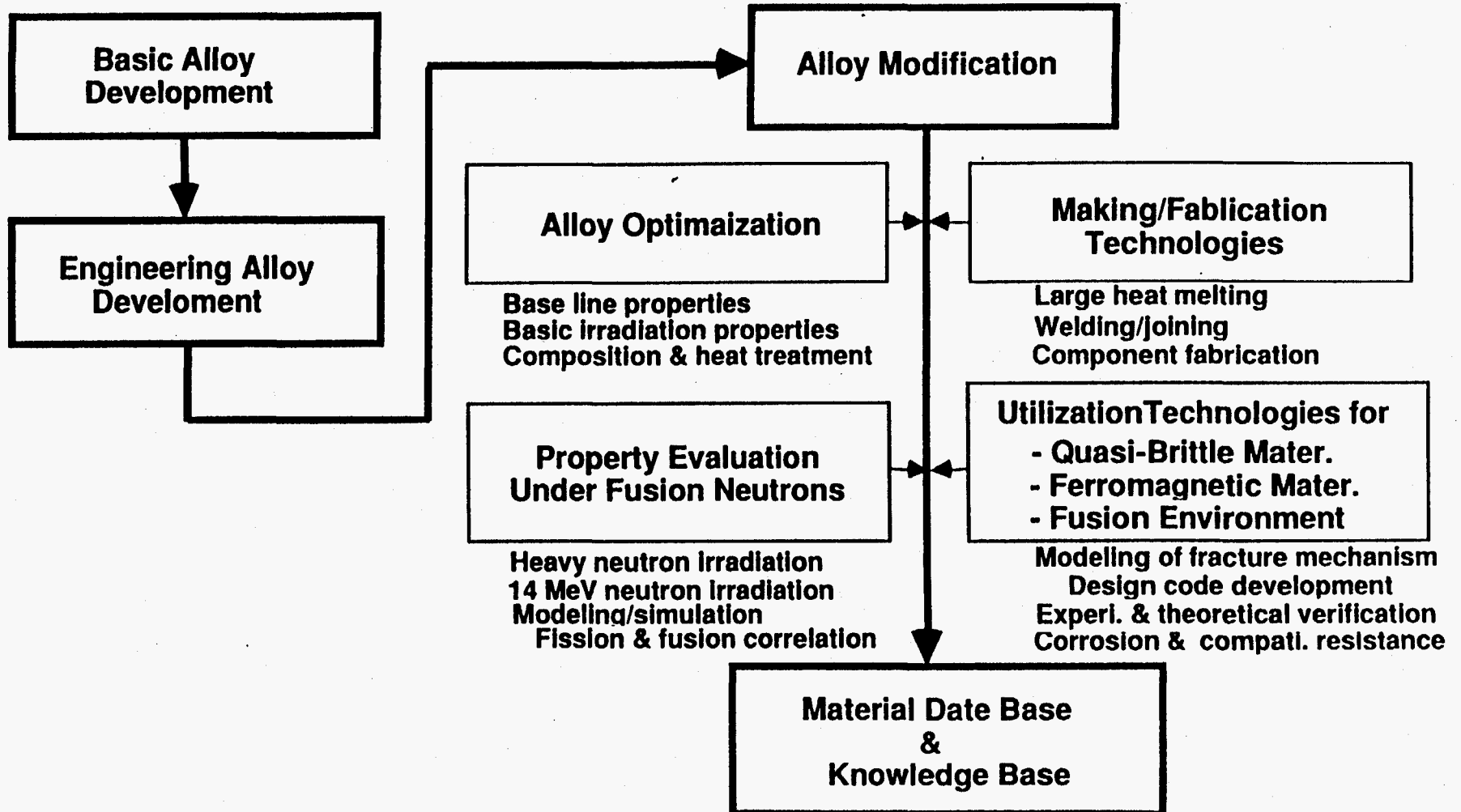
**IEA Workshop on LAM  
Culham, U.K.  
October 24, 25, 1996**

**JAERI**

## Time Schedule of F82H Development for Fusion Device

	1995	2000	2005	2010	2015	2020	2025	2030	2035	2040
ITER/Prototype/Demo Reactor	(ITER) Design/Construction			BPP	APP					
				(Prototype)	Design			Construction	Operation	
IFMIF	CDA EDA Construction (C&R)						Operation			
(1)Material Development - Standard/Control Properties - Basic Irradiation Properties - Compositional Optimization	IEA Collaboration/Round Robin Tests HFIR Phase3									
(2)Making/Fabrication Technologies - Large Heat Melting - Welding/Joining - Component Fabrication	5t	30t	30t	100t	TIG/EB/HIP Blanket Module, etc.					
(3)Properties under Fusion Neutrons - Heavy Irradiation - 14MeV Neutron Irradiation - Fission & Fusion Correlation	SSTT Fission Reactor (HFIR/Phenix/JOYO) Irradiation IFMIF Irradiation Model Simulation									
(4)Utilization Technologies for - Quasi-Brittle Materials  - Ferromagnetic Materials - Fusion Environment	Fracture Mechahism Design Code JFT-2M Coating, etc.									
(5)Data Accumulation for Designing & Construction	Module Test using ITER JOYO/IFMIF Irradiation									

# LOW ACTIVATION FERRITIC STEEL DEVELOPMENT



# **Material/Plasma Compatibility Experiment (JFT-2M)**

## **OBJECTIVE**

Find the Problems and Solutions for the Prototype Fusion Reactor using Ferritic Steel as a Structural Material

## **EXPERIMENT**

1. Simulation of Ferritic Steel Vacuum Vessel (Figure 1)
  - Whole Inner Surface to be covered by F82H except for the Access Ports
  - Estimate the Error Magnetic Field  
(Other Options)
    - Plate Arrangement for the Minimum Affection of Port Opening
    - Plate Arrangement to realize Ripple-less Mode
2. Simulation of Ferritic Steel Blanket Module (Figure 2)
  - Ferritic Steel Blanket Module (simulating SSTR Blanket Module; Figure)
  - Estimate the Error Magnetic Field

## TEST ITEMS

### *Plasma Generation/Control*

- (1) Firing Properties
- (2) Plasma Control ability (Behavior of Magnetic Sensor)
- (3) Prevention from Locked Mode due to Error Magnetic Field
- (4) Effect of Error Field to Shut-up Properties (H mode, High  $\beta_p$ , etc.)
- (5) Simulation of Prototype Reactor Module  
(Plasma Geometry, Blanket, etc.)
- (6) Elimination of Toroidal Ripple
- (7) Flashing Cleaning on Ferritic Steel
- (8) Impurity Control and Retention on Ferritic Steel

### *Material Properties*

- (1) Mechanical/Physical Properties of F82H under High Magnetic Field
- (2) Surface Properties of F82H in Plasma Environment
- (3) Out-gas Properties of F82H
- (4) Establishment of Large Heat Production Technique ( $\sim 30$  tons) of F82H

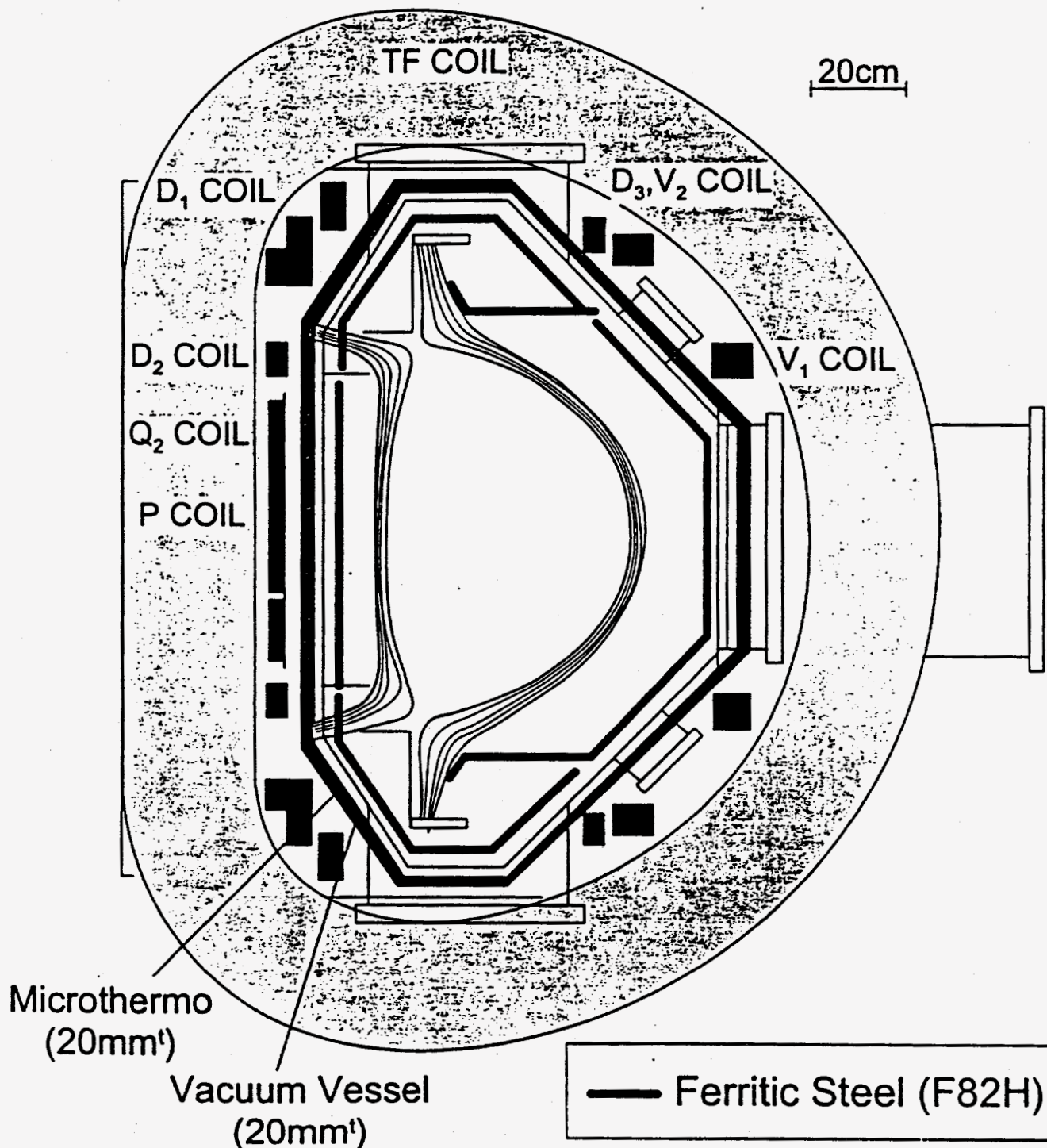


Figure 1 Simulation of Ferritic Steel Vacuum Vessel

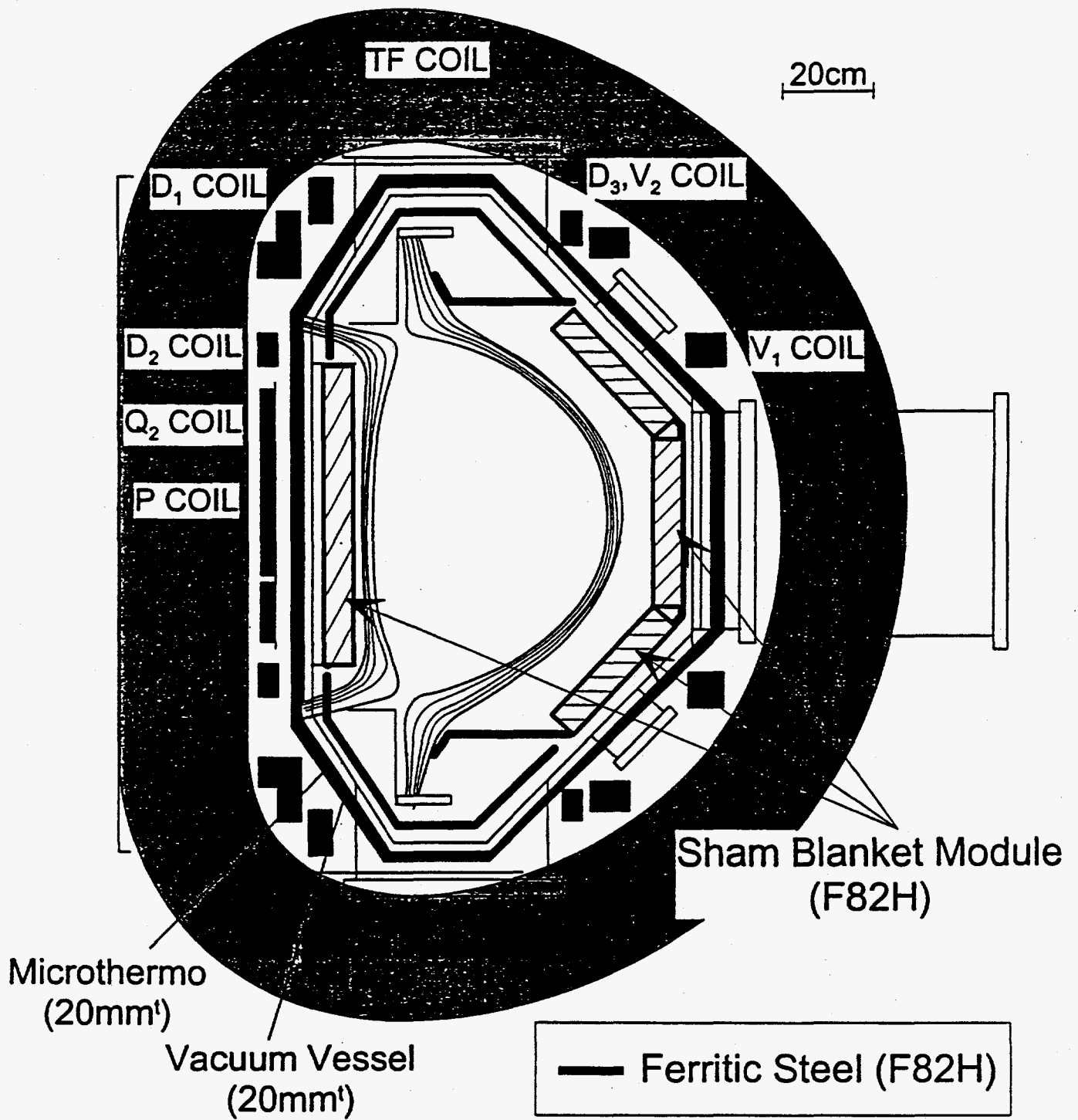


Figure 2 Simulation of Ferritic Steel Blanket Module



*IEA Workshop and Working Group Meeting  
on Ferritic Steels  
JET Headquarters, Culham, UK  
24-25 October, 1996*

## **Present Status and the Future Plan on JLF-1 and the Japanese University Activities**

**AKIRA KOHYAMA**  
**Institute of Advanced Energy, Kyoto University,**  
**Gokasho Uji, Kyoto 611, Japan**  
**e-mail: [kohyama@iae.kyoto-u.ac.jp](mailto:kohyama@iae.kyoto-u.ac.jp)**





## **MAJOR RESEARCH SITES ON Low Activation Ferritic Steels for Fusion Applications**

<b>Hokkaido Univ.</b>	<b>H. Takahashi, S. Ohnuki, S. Watanabe</b>
<b>Muroran Int. Tech.</b>	<b>T. Misawa</b>
<b>Tohoku Univ.</b>	<b>H. Kayano, A. Kimura, H. Kurishita, T. Shibayama, T. Yamamoto, M. Narui</b>
<b>Univ. of Tokyo</b>	<b>Y. Kohno, K. Asakura, M. Uesaka, K. Morishita,</b>
<b>Seikei Univ.</b>	<b>M. Isogo</b>
<b>Tokyo Inst. Tech.</b>	<b>A. Wada</b>
<b>NIFS</b>	<b>T. Muroga, A. Nishimura, K. Nishimura</b>
<b>Kyoto Univ.</b>	<b>A. Kohyama, Y. Kato, Y. Takeuchi</b>
<b>NRIM</b>	<b>F. Hasegawa, K. Noda</b>
<b>JAERI</b>	<b>A. Hishinuma, K. Shiba, S. Jitsukawa</b>
<b>Nippon Steel Corp.</b>	<b>T. Hasegawa, H. Mabuchi</b>
<b>Nippon Kokan</b>	<b>M. Tamura</b>



## MAJOR RESEARCH ACTIVITIES ON JLF-1 QUALIFICATION

### *Magnetic properties:*

M. Uesaka (U. Tokyo), M. Isogo (Seikei U.)

### *Mechanical properties:*

*Charpy*; Y. Kohno (U. Tokyo), H. Kurishita (Tohoku U.)

*Fracture Toughness*; A. Nishimura (NIFS)

*Tensile*; A. Kohyama, Y. Katoh (Kyoto U.), A. Kimura (Tohoku U.)

*Fatigue*; K. Morishita, K. Miya (U. Tokyo)

*Creep*; K. Asakura (U. Tokyo), T. Hasegawa (NSC)

### *Welding process optimization:*

T. Hasegawa (NSC), A. Kohyama (Kyoto U.)

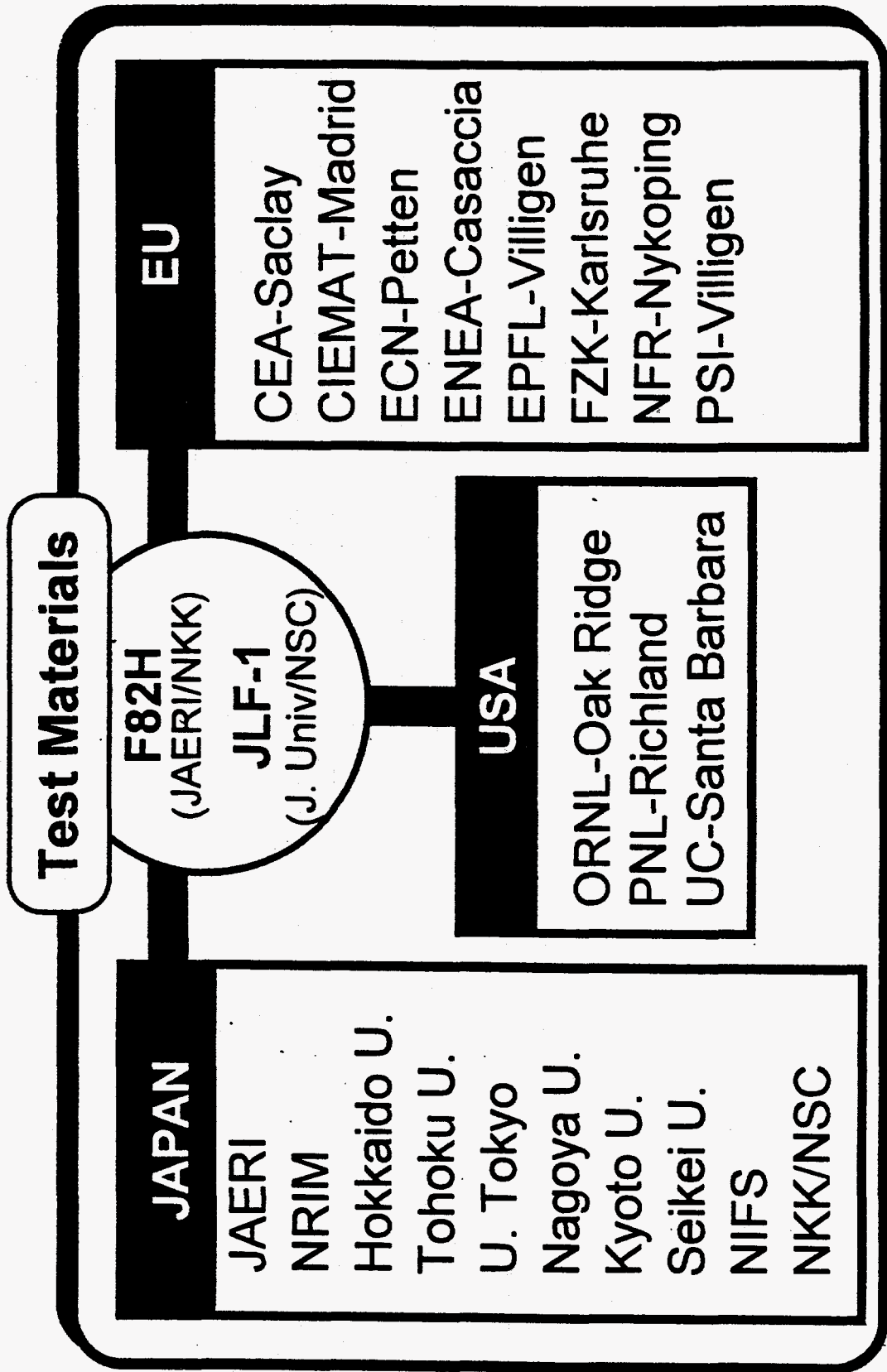
### *Design Code:*

A. Wada (TIT), K. Miya (U. Tokyo)

### *Compatibilities with environment:*

T. Misawa (MIT), A. Kimura (Tohoku U.)

# IEA Collaborative Experiments on Low Activation Ferritic Steels



*Duration: 1994-1999*

## JLF-1 (2nd heat: IEA heat)

### 15mm thick plates

#	Plate ID	Plate size	Responsibility	Weld/Heat treatment	Experimental
1	HEAT2-1-1-1	15x340x450	ORNL	As NT	ORNL-DCT, 1/3-CVN, Full-CVN etc.
2	HEAT2-1-1-2	15x340x450	ORNL	TIG/PWHT	ORNL-DCT, 1/3-CVN, Full-CVN etc.
3	HEAT2-1-1-3	15x340x450	ORNL	As PWHT, EB	ORNL-DCT, 1/3-CVN, Full-CVN etc.
4	HEAT2-1-1-4	15x340x450	PNNL	As NT, As PWHT	Microstructural identification etc.
5	HEAT2-1-1-5	15x340x450	EU	As NT	Fatigue, Toughness, Creep
6	HEAT2-2-1-1	15x340x450	EU	As NT	Precipitation identification
7	HEAT2-2-1-2	15x340x450	EU	As PWHT	Fatigue, Toughness, Creep, Microstructural inspection
8	HEAT2-2-1-3	15x340x450	EU	TIG/PWHT	Fatigue, Toughness, Creep
9	HEAT2-2-1-4	15x340x450	EU	TIG/PWHT	Precipitation identification
10	HEAT2-2-1-5	15x340x450	KU	As NT	Baseline mechanical properties
11	HEAT2-3-1-1	15x340x450	KU	TIG/PWHT	Baseline mechanical properties
12	HEAT2-3-1-2	15x340x450	KU	EB/PWHT	Baseline mechanical properties
13	HEAT2-3-1-3	15x340x450	KU(reserve)	As NT	Reserve
14	HEAT2-3-1-4	15x340x450	KU(reserve)	As NT	Reserve
15	HEAT2-3-1-5	15x340x450	KU(reserve)	As PWHT	Reserve
16	HEAT2-4-1-1	15x340x450	KU(reserve)	As PWHT	Reserve
17	HEAT2-4-1-2	15x340x450	KU(reserve)	TIG/PWHT	Reserve
18	HEAT2-4-1-3	15x340x450	KU(reserve)	TIG/PWHT	Reserve
19	HEAT2-4-1-4	15x340x450	KU(reserve)	EB	Reserve
20	HEAT2-4-1-5	15x340x450	UT/Tohoku	As NT	Magnetic properties, Creep rupture etc.
21	HEAT2-5-1-1	15x340x450	UT/Tohoku	As PWHT, TIG/PWHT	Magnetic properties, Creep rupture etc.
22	HEAT2-5-1-2	15x340x450	NIFS	As NT	Chemical properties (compatibility) etc.
23	HEAT2-5-1-3	15x340x450	NIFS	TIG/PWHT	Chemical properties (compatibility) etc.
24	HEAT2-5-1-4	15x340x450	NSC	As NT, As PWHT	Base material/Baseline properties
25	HEAT2-5-1-5	15x340x450	NSC	TIG/PWHT	Welded joints/Baseline properties

### 25mm thick plates

#	Plate ID	Plate size	Responsibility	Heat treatment	Experimental
1	HEAT2-1-2-1	25x340x500	ORNL/PNNL	As NT	Full-DCT, Full-CVN etc.
2	HEAT2-1-2-2	25x340x500	ORNL	TIG/PWHT	Full-DCT, Full-CVN etc.
3	HEAT2-1-2-3	25x340x500	EU	As NT	Fatigue, Toughness, Creep, Microstructural inspection
4	HEAT2-2-2-1	25x340x500	EU	TIG/PWHT	Fatigue, Toughness, Creep, Microstructural inspection
5	HEAT2-2-2-2	25x340x500	KU	As NT	Baseline mechanical properties
6	HEAT2-2-2-3	25x340x500	KU	As PWHT	Baseline mechanical properties
7	HEAT2-3-2-1	25x340x500	KU	TIG/PWHT	Baseline mechanical properties
8	HEAT2-3-2-2	25x340x500	KU(reserve)	As NT	Reserve
9	HEAT2-3-2-3	25x340x500	KU(reserve)	As PWHT	Reserve
10	HEAT2-4-2-1	25x340x500	KU(reserve)	TIG/PWHT	Reserve
11	HEAT2-4-2-2	25x340x500	KU(reserve)	TIG/PWHT	Reserve
12	HEAT2-4-2-3	25x340x500	UT/Tohoku	TIG/PWHT	Magnetic properties, Creep rupture etc.
13	HEAT2-5-2-1	25x340x500	NIFS	As PWHT	Chemical properties (compatibility) etc.
14	HEAT2-5-2-2	25x340x500	NSC	As NT, As PWHT	Base material/Baseline properties
15	HEAT2-5-2-3	25x340x500	NSC	TIG/PWHT	Welded joints/Baseline properties

ORNL/ R.L. Klueh  
PNNL/ D.S. Gelles  
EU/ K. Ehrlich

KU (Kyoto U.)/ A. Kohyama  
UT (U. Tokyo)/ Y. Kohno, K. Asakura, M. Uesaka, K. Morishita  
Tohoku (Tohoku U.)/ A. Kimura, T. Shibayama  
NIFS/ A. Nishimura, Y. Katoh, T. Muroga  
NSC (Nippon Steel Corp.)/ T. Hasegawa, H. Mabuchi



JAPAN-USA COLLABORATION



1995-2000

**for FUSION MATERIALS**

# The Monbusho/US-DOE Collaboration for Fusion Materials

## - JUPITER -

Ver.7 :6/10/96

### HFIR

In-situ Resistivity Measurement (1)

Advanced irradiation Experiment

High DPA irradiation Experiment

Low DPA/High T irradiation Experiment

Varying Temperature Experiment

Advanced irradiation Experiment

1995	1996	1997	1998	1999	2000
3/6/96	■ P3-1: 300-600C	(TRIST-ER-1)		TRIST Irradiation	6 cycles (3dpa for Ceramics)
(6/25/96) 8/2/96	■ P3-2: 300C (HFIR-MFE-RB-11J) ■ P3-3: 500C (HFIR-MFE-RB-12J) P3-4: 500C, 800C			Irradiation at RB* position with thermal shielding Irradiation at RB* position	10 cycles (5dpa for Fe)
			■ P3-5: 200C, 300C, 400C, 500C		Irradiation at PTP test matrix by 6/96 10 cycles
		■ P3-6: 500C, 800C, 1000C			Irradiation at PTP test matrix by 6/96 TBD
			■ P3-7: 400-600C		test matrix by 6/96 TBD
				□ P3-8: 200C - 800C ■ P3-9: 300C, 500C	TBD

ISI Outage →

### HFBR

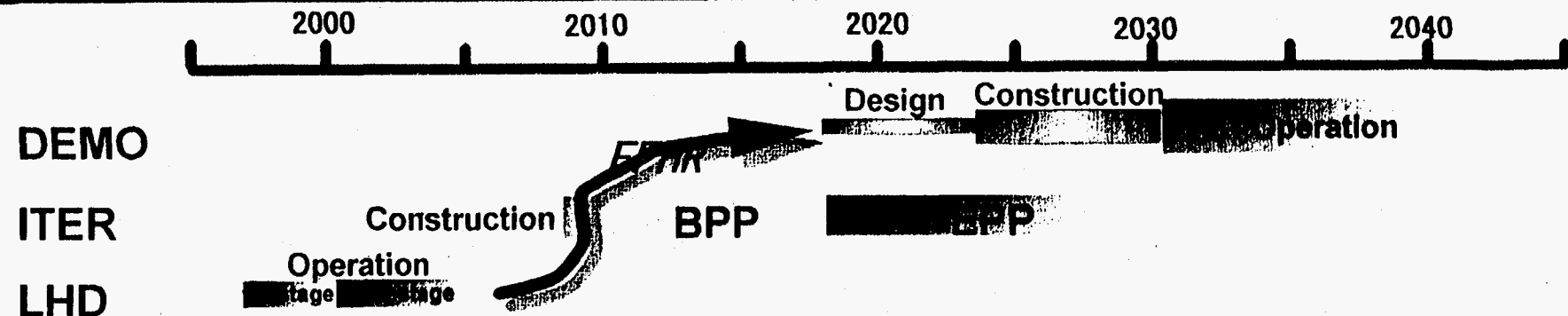
■ P3-H1: 200C, 300C (ISEC-3)

### ATR

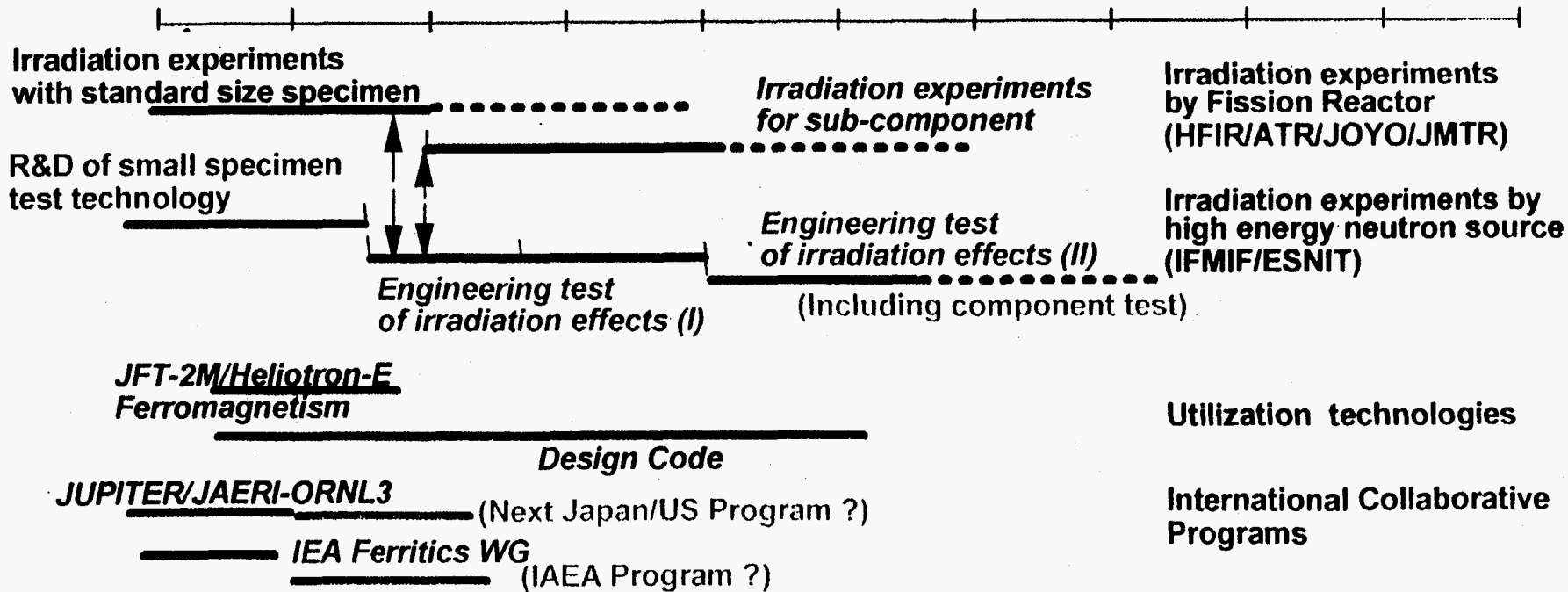
12/2/95	□ P3-A1: 200C, 300C				(5dpa for V)	5 cycles
		□ P3-A2: 400C, 600C	feasibility check by 6/96			TBD



# Fusion Energy Development Plan & LAFs R&D



## Ferritic steel R&D



U. S. FERRITIC STEEL PROGRAM

R. L. Klueh

Oak Ridge National Laboratory  
Oak Ridge, Tennessee USA

IEA Working Group Meeting on Ferritic Steels for Fusion

Culham, UK

19-20 September 1995

## GENERAL STATUS OF U.S. FUSION PROGRAM

R. L. Klueh

- Budget for FY 1996: \$240M
- Fusion Advanced Materials Program budget FY 1996: \$8M
- Expected budget for FY 1997: \$220M
- Expected Fusion Advanced Materials Program budget FY 1996: \$5M



## U. S. PROGRAM WILL EMPHASIZE FRACTURE STUDIES

- Unirradiated studies
  - Micromechanics of fracture
  - Size effects/crack geometry effects, advanced fracture mechanics
  - Low-cycle fatigue
  - Sub-critical crack growth
  - Composition optimization
  - Thermal stability
  
- Irradiated studies
  - Tensile, Charpy, compact tension, and TEM
  - Microstructure-hardening-fracture correlations
  - Helium effects

## U. S. FERRITIC STEEL PROGRAM -- THE FUTURE

- Budget uncertainty has hindered program planning
- Ferritic steels in the U. S. program are not top priority
- U. S. program expects to put less emphasis on ferritic steels than either Japan or Europe
- Essentially all of U. S. work will be part of the IEA collaboration
- Irradiation experiments in the U. S. program will be part of collaboration between the U. S. and Japan -- JAERI and Monbusho
- No irradiation experiments are presently planned beyond those in the two Japan collaborations
- Two HFIR RB\* capsules are being built for the Japan/U.S. collaboration for insertion in FY 1997
- Two HFIR target capsules containing tensile and Charpy specimens were irradiated in FY 1995-1996, and the specimens are ready for testing

PROPOSED SCHEDULE OF WORK ON IEA HEATS

Work Period	Task	Remarks
FY 1995-1996	Characterization of IEA and JLF-1 Heats.	TEM, metallography, mechanical properties, etc.
FY 1995-1996	Low-cycle fatigue.	Unirradiated properties.
FY 1995-1996	Mixed-mode fracture toughness and hydrogen effects.	Unirradiated properties determined on F82H (non-IEA heat) to be extended to IEA heat of F82H.
FY 1997	Begin irradiation of HFIR RB* capsules at 300 and 500°C.	Capsules are joint U.S./Japan collaborations.
FY 1996	Test F82H irradiated in FFTF and EBR-II to >50 dpa at 420°C and 30 dpa at 375°C.	Different heat of F82H; TEM, tensile, Charpy, and fracture toughness specimens are available for testing.
FY 1996-1998	Characterize IEA heat to determine fracture behavior in unirradiated condition.	Develop "valid" K(T) curves with "standard" specimens and baseline curves for subsized specimens, etc.
FY 1996-1998	Complete irradiation of HFIR target capsules. Test specimens.	Capsules irradiated at 300 and 400°C to 10-13 dpa contain IEA heat of modified F82H along with other steels.

PROPOSED SCHEDULE OF WORK ON IEA HEATS (CONTINUED)

Work Period	Task	Remarks
FY 1997-1998	Complete irradiation of RB* capsules and test mechanical properties specimens.	Irradiations to 5 dpa.
FY 1997-1998	Thermal stability studies.	Aging to 20,000 h.
FY 1997-1998	Analyze data from two RB* experiments.	Determine irradiation-induced shifts and curve-shape changes for IEA F82H and JLF-1 base metal, other TMTs, welds, etc.
FY 1998-1999	Composition optimization studies.	Studies based on properties observations of IEA and other heats.
FY 1997-1999	Microstructural and micromechanical experiments.	Complement mechanical testing with micromechanical (e.g., confocal microscopy) and microstructural (TEM/AEM, SANS, AP) experiments and models (FEM/local fracture).
FY 1998-2000	Complete characterization of fracture behavior of IEA F82H and extend work to welds, JLF-1, and other heats.	Correlate irradiation-induced shifts and curve shape changes in K(T) with changes in constitutive and local fracture properties, etc.

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