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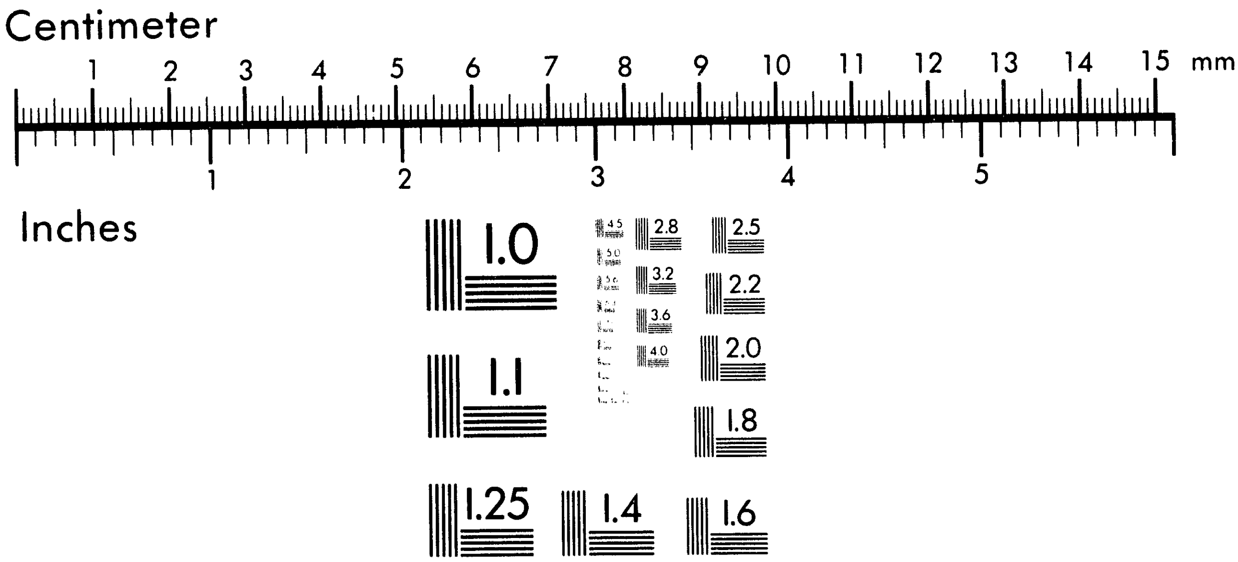

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IRRADIATION ASSISTED STRESS CORROSION CRACKING OF HTH ALLOY X-750 AND ALLOY 625

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NOTICE

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IRRADIATION ASSISTED STRESS CORROSION CRACKING OF HTH ALLOY X-750 AND ALLOY 625

ABSTRACT

In-reactor testing of bolt-loaded precracked compact tension specimens was performed in 360°C water to determine the effect of irradiation on the SCC behavior of HTH Alloy X-750 and direct aged Alloy 625. Out-of-flux and autoclave control specimens were also tested to provide baseline data. The primary test variables were stress intensity factor (K_I), fluence, chemistry, processing history and prestrain. Results for the first series of experiments were presented at the Sixth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems which was held in San Diego, CA. Data from two more recent experiments are compared with the previous results. The recent tests confirm that high irradiation levels significantly reduce SCC resistance in HTH Alloy X-750. Heat-to-heat differences in IASCC were related to differences in boron content, with low boron heats showing improved SCC resistance. The in-reactor SCC performance of Alloy 625 was superior to that for Alloy X-750, as no cracking was observed in any Alloy 625 specimens even though they were tested at very high K_I and fluence levels.

A preliminary SCC usage model developed for Alloy X-750 indicates that in-reactor creep processes, which relax stresses but also increase crack tip strain rates, and radiolysis effects accelerate SCC. Hence, in-reactor SCC damage under high flux conditions may be more severe than that associated with postirradiation tests. In addition, preliminary mechanism studies were performed to determine the cause of IASCC in Alloy X-750.

Specific topics discussed in this presentation are given below:

- Summary of High Temperature Stress Corrosion Cracking (HTSCC) and Low Temperature Crack Propagation (LTCP)
- Test Materials and Experimental Procedure
- New Test Results Supporting Conclusions Presented at 6th Int'l Symposium on Environmental Degradation
- Comparison of In-Reactor and Postirradiation SCC Testing
- IASCC Mechanisms

HIGH AND LOW TEMPERATURE SCC BEHAVIOR OF HTH ALLOY X-750 AND ALLOY 625

HTH Alloy X-750 (1093°C solution anneal for 1-2 h followed by 20 h age at 704°C)

Relatively good HTSCC resistance.
Superiority of HTH over Conditions AH and BH attributed to :
- $M_{23}C_6$ along grain boundaries
- Large grain size
- Reduction of intergranular segregation of P & S

Susceptible to LTCP below 150°C in PWR water when cracks are present.
$K_{ISCC\text{-}LT}$ is less than 50% of K_{IC}.

Potential SCC sequence for Alloy X-750:
- SCC initiation at high temperature
- SCC growth at high temperature to critical flaw size ($K_{ISCC\text{-}LT}$)
- LTCP and complete fracture during cooldown.

Alloy 625 (direct aged at 663°C for 80 h)

Good resistance to HTSCC

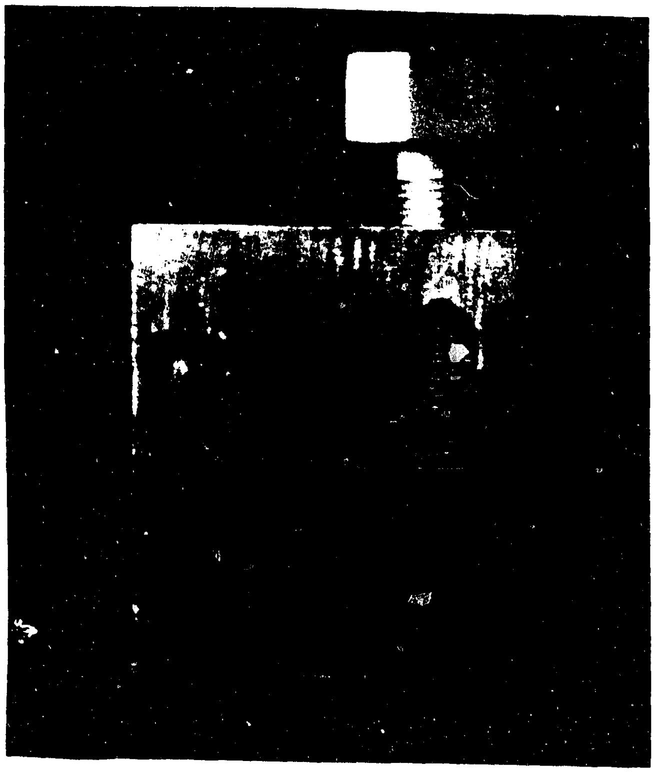
Virtually immune to LTCP
- Superiority associated with reduced hydrogen absorption at crack tip.

Chemical Composition Of Test Materials (in weight percent)

Heat*	Material	Ni	Cr	Fe	Ti	Cb	Al	C	Mo	P	S	B
					ALLOY X-750							
A1	HTH	71.83	15.27	8.05	2.61	0.97	0.75	0.041	0.02	0.002	0.0003	0.0028
A2	HTH	71.88	15.54	7.98	2.65	1.05	0.68	0.043	0.02	0.002	0.0001	0.0027
A3	HTH	71.30	15.58	8.15	2.61	1.10	0.69	0.042	0.13	0.003	0.0001	0.0020
A4	HTH	72.93	15.12	6.81	2.69	1.04	0.70	0.044	0.03	0.001	0.0005	0.0022
A4b	HTH	73.41	15.22	6.84	2.71	1.06	0.71	0.043	0.03	0.001	0.0005	0.0022
A5	HTH	71.62	15.35	7.95	2.50	1.01	0.74	0.051	0.01	0.004	0.001	0.0024
A6	HTH	70.70	15.04	7.90	2.58	1.01	0.75	0.040	0.01	0.001	0.001	0.0026
A7	HTH	72.27	15.83	8.06	2.59	1.01	0.67	0.039	0.01	0.003	0.003	0.0002
A8	HTH	71.55	15.53	7.65	2.67	1.02	0.75	0.042	0.01	0.004	0.001	<0.001
A9	HTH	71.22	15.49	7.70	2.66	1.02	0.73	0.037	0.01	0.004	0.003	<0.001
A10	HTH	71.17	15.46	8.33	2.67	1.00	0.77	0.072	0.01	0.007	0.001	0.0070
A11	HTH	71.86	15.46	7.91	2.69	0.99	0.78	0.054	0.01	0.003	0.003	0.0090
A13	HTH	71.11	15.46	8.26	2.61	0.99	0.82	0.066	NA	0.004	0.001	NA
B1	HTH	71.73	15.52	8.41	2.42	0.89	0.63	0.05	NA	0.005	0.005	0.0045
					ALLOY 625							
A12	A625	62.19	20.59	4.17	0.33	3.64	0.33	0.033	8.51	0.005	0.0011	0.002
D1	A625	60.98	21.82	3.65	0.25	3.47	0.25	0.039	9.04	0.006	0.001	0.001
D2	A625	60.03	21.97	4.40	0.27	3.53	0.26	0.022	9.01	0.008	0.001	0.001

*The letter denotes Vendor A, B or D.

BOLT-LOADED COMPACT TENSION SPECIMEN
(Width = 20 mm, Thickness = 10 mm)



Precracked compact tension (CT) specimens were irradiated in a test reactor at about 360°C. All specimens were exposed to water containing 40-60 cc H_2/kg H_2O during normal reactor operations above 175°C and a lower level of 17 cc H_2/kg H_2O or less at temperatures below 175°C to minimize the potential for LTCP. The room temperature pH of the water was between 10.1 and 10.3, oxygen was less than 100 ppb and conductivity was betwee 21 and 85 μS/cm.

CONCLUSIONS FROM PAPER AT 6th INT'L SYMPOSIUM ON ENVIRONMENTAL DEGRADATION (San Diego) ARE SUPPORTED BY NEW TEST RESULTS

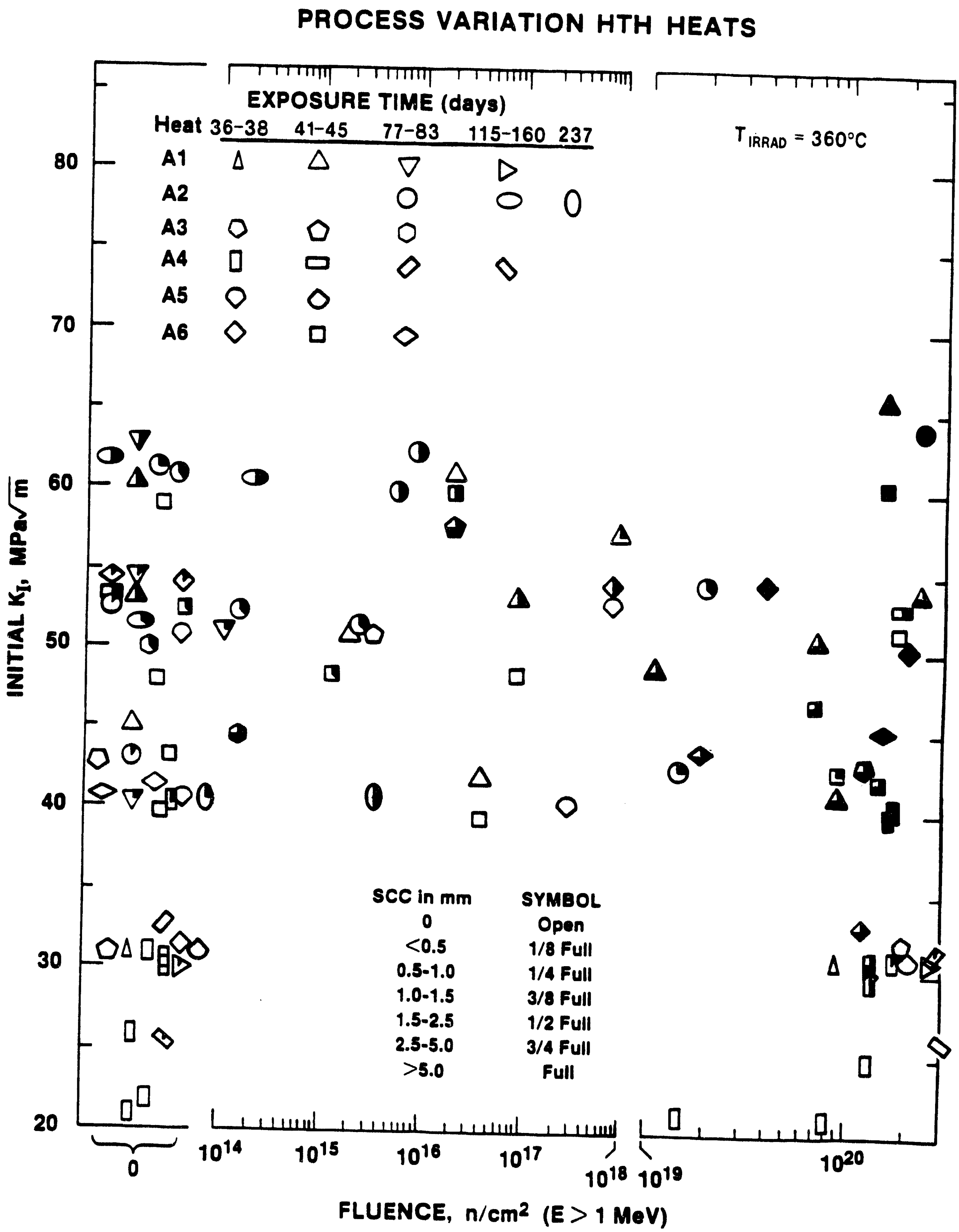
High irradiation levels significantly reduce SCC resistance in HTH Alloy X-750. IASCC at high fluences attributed primarily to irradiation-induced microstructural changes.

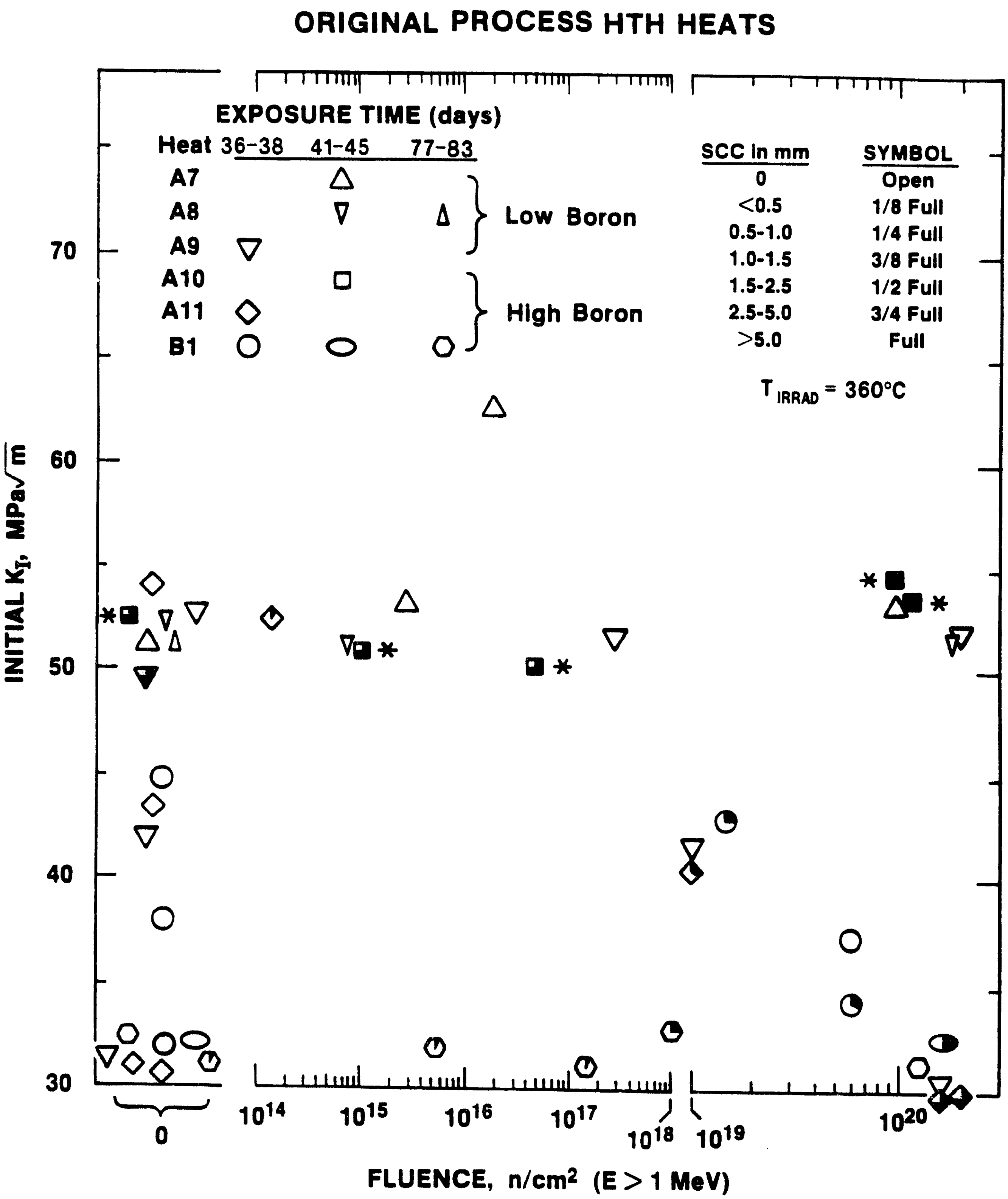
HTH X-750 shows a small acceleration in cracking at low fluences (10^{14} to 10^{18} n/cm^2) that was attributed to radiolysis effects.

Heat-to-heat differences in IASCC correlated with differences in B content. Heats with less than 10 ppm B showed little or no effect of irradiation at both high and low temperatures.

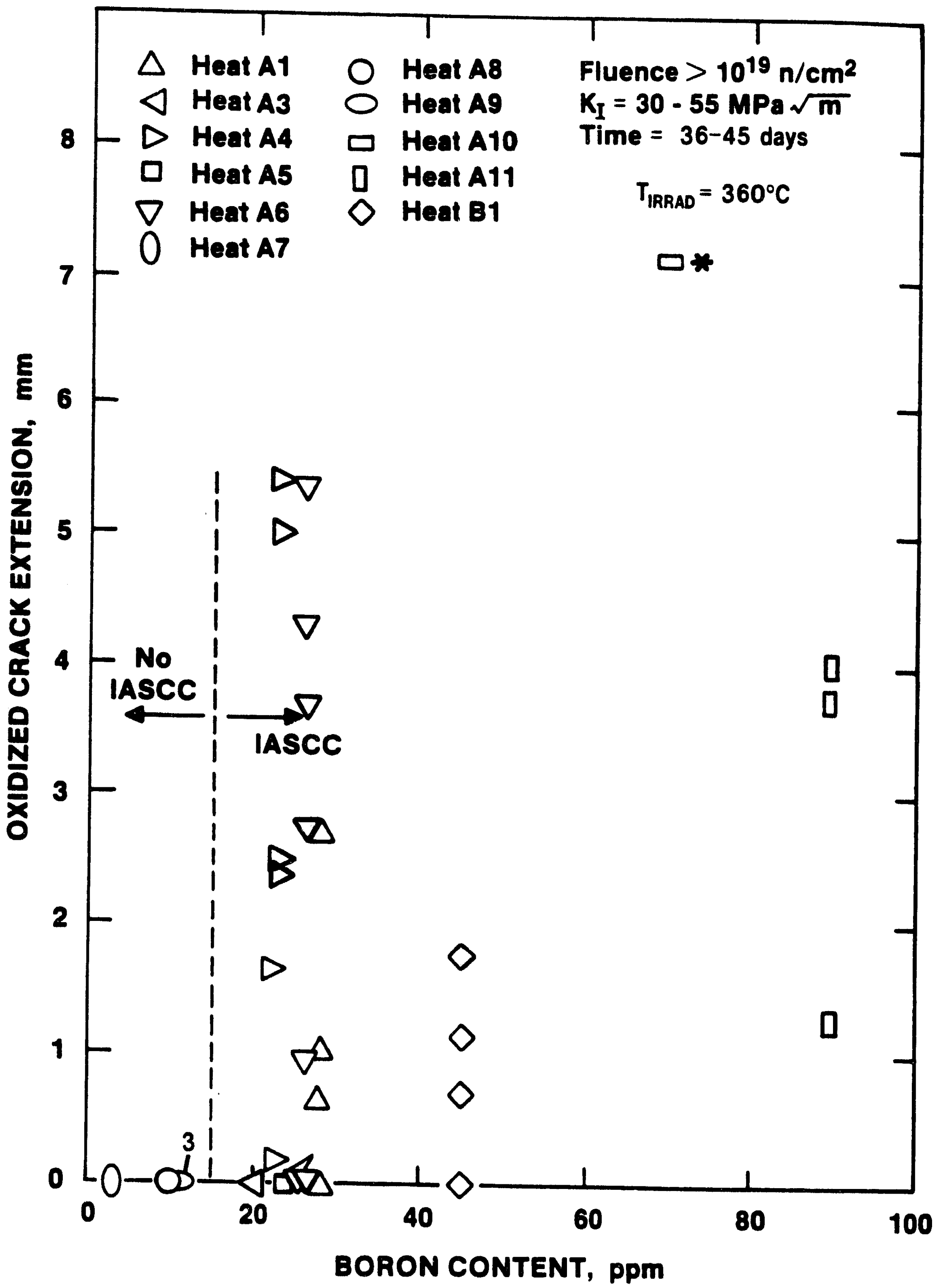
No evidence of SCC was observed in any precracked Alloy 625 specimens despite being highly loaded and irradiated.

IASCC2

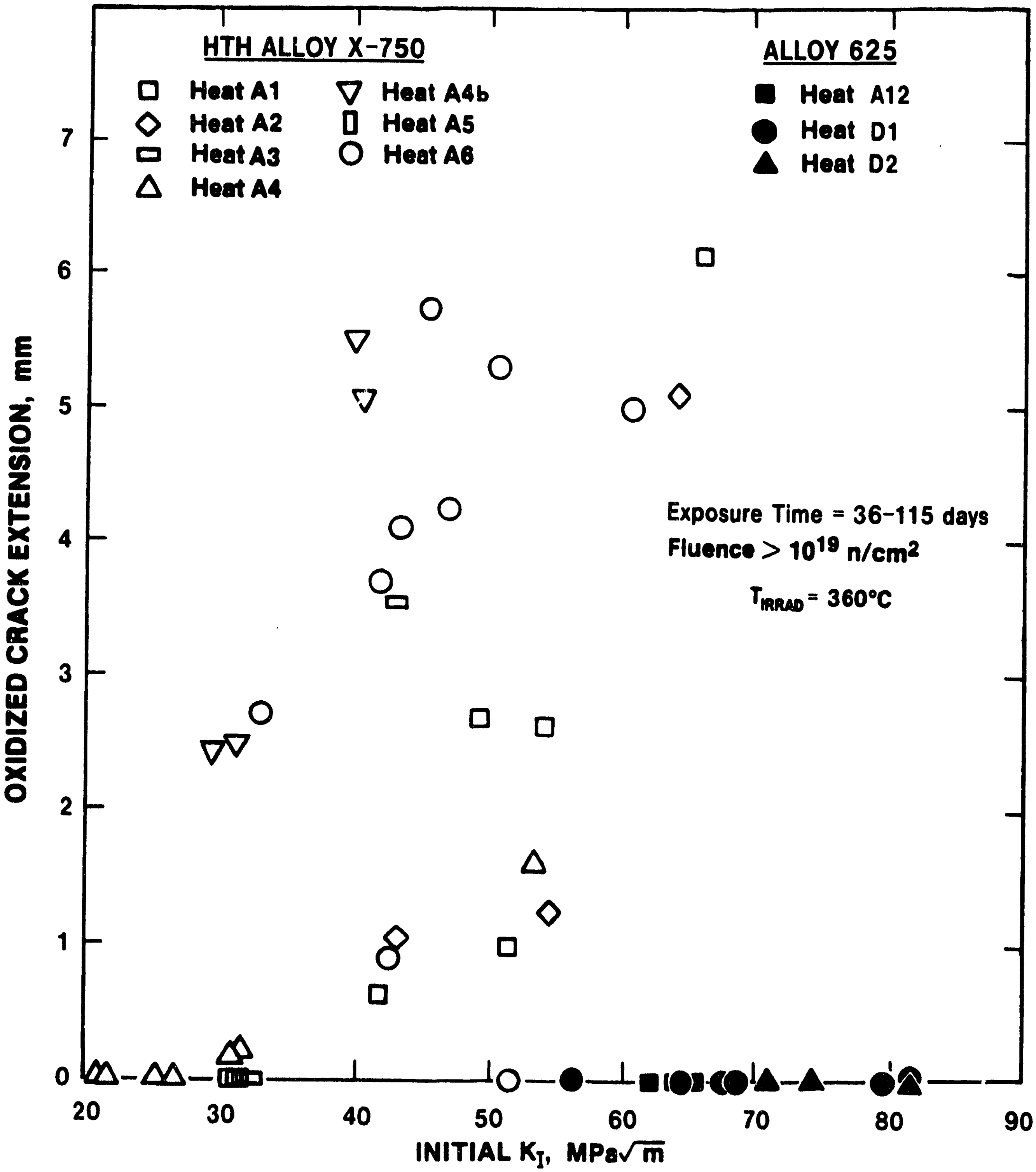

PROCESS VARIATION HTH HEATS
EXPOSURE TIME (days)
Heat 36-38 41-45 77-83 115-160 237
A1
A2
A3
A4
A5
A6
T IRRAD = 360°C
INITIAL K I, MPa√m
80
70
60
50
40
30
20
SCC in mm
0
<0.5
0.5-1.0
1.0-1.5
1.5-2.5
2.5-5.0
>5.0
SYMBOL
Open
1/8 Full
1/4 Full
3/8 Full
1/2 Full
3/4 Full
Full
0
10^14
10^15
10^16
10^17
10^18
10^19
10^20
FLUENCE, n/cm² (E > 1 MeV)



Asterisk denotes probable low temperature crack propagation during interim cooldown.


Heat A1
Heat A3
Heat A4
Heat A5
Heat A6
Heat A7
Heat A8
Heat A9
Heat A10
Heat A11
Heat B1
Fluence > 10^19 n/cm^2
K_I = 30 - 55 MPa √m
Time = 36-45 days
T_IRRAD = 360°C
No IASCC
IASCC
3
OXIDIZED CRACK EXTENSION, mm
BORON CONTENT, ppm
0
1
2
3
4
5
6
7
8
20
40
60
80
100

COMPARISON OF IASCC RESISTANCE FOR ALLOY 625 AND HTH ALLOY X-750 PROCESS VARIATION HEATS



Summary of Test Conditions for Precracked Alloy 625 Specimens. No SCC Was Observed in Any Specimens.

Heat	Fluence (n/cm^2)	K_I-initial ($MPa\sqrt{m}$)	Time (days)
A12	0	64-66	45-83[a]
	0	62	160
	3.2×10^{14}	64	83
	9.8×10^{17}	62	160
	$18\text{-}28 \times 10^{19}$	64-65	45-83[b]
D1	0	43-65	77[c]
	0	63	237
	$1.5\text{-}87 \times 10^{14}$	53-64	77[c]
	24×10^{14}	63	237
	$2.1\text{-}7.0 \times 10^{19}$	56-69	77[b]
	$23\text{-}44 \times 10^{19}$	65-81	77[c]
D2	0	67	77
	$2.2\text{-}3.8 \times 10^{14}$	56-70	77[b]
	3.1×10^{19}	71	77
	$18\text{-}28 \times 10^{19}$	74-81	77[a]

[a] 2 Specimens [b] 3 Specimens [c] 4 Specimens

IN-REACTOR VS. POSTIRRADIATION SCC TESTING

Postirradiation Controlled Load	In-Reactor Constant Displacement	In-Reactor Controlled Load
Not prototypic flux	Accelerated & prototypic flux	Accelerated & prototypic flux
No radiolysis No in-reactor creep	Prototypic and accelerated radiolysis. In-reactor creep may cause loads to decrease more rapidly than experienced by components.	Prototypic and accelerated radiolysis. Controlled loading can provide prototypic loadings by offsetting excess in-reactor creep.
Specimens are loaded after irradiation hardening occurs, leading to excessively high stresses ahead of crack tip.	Load path history is similar to that experienced by components.	Load path history can be controlled, so it is prototypic of actual components.
Instrumented tests provide data supporting SCC model development & confirmation.	Tests are not instrumented. Periodic inspections must be performed to determine extent of cracking.	Instrumented tests provide data supporting SCC model development & confirmation.

USAGE MODEL FOR PREDICTING IASCC INCUBATION TIME

OBJECTIVE

To account for the mitigating effect of stress relaxation and the aggravating effect of irradiation damage on SCC incubation time (t_{INC})

USAGE CONCEPT

SCC occurs when Usage (U) = 1.0

Assumptions:
1) Instantaneous SCC usage rate depends on instantaneous K_I
   (Note: "Instantaneous K_I" is treated as if derived by a monotonically increasing load.)
2) SCC usage rate increases with increasing fluence

REQUIRED DATA

K_I dependency on crack incubation time
Rate of stress relaxation
Relative usage rate for irradiation damage

In-Reactor crack incubation time at fluence of $\sim 2 \times 10^{20}$ n/cm^2

IRRADIATION DAMAGE FACTOR

ASSUMPTIONS:

Based on postirradiation test results, a <u>33-fold reduction</u> in SCC incubation time was assumed at 2 x 10^{20} n/cm^2.

This is judged to be a very conservative assumption because applying loads after irradiation results in significantly higher stresses ahead of the crack due to irradiation-induced strengthening.

About a <u>2-fold reduction</u> in SCC incubation time was assumed at 1 x 10^{18} n/cm^2.

This is judged to be a conservative assumption because crack extension values at fluences up to 1 x 10^{18} n/cm^2 were less than twice those for out-of-flux specimens.

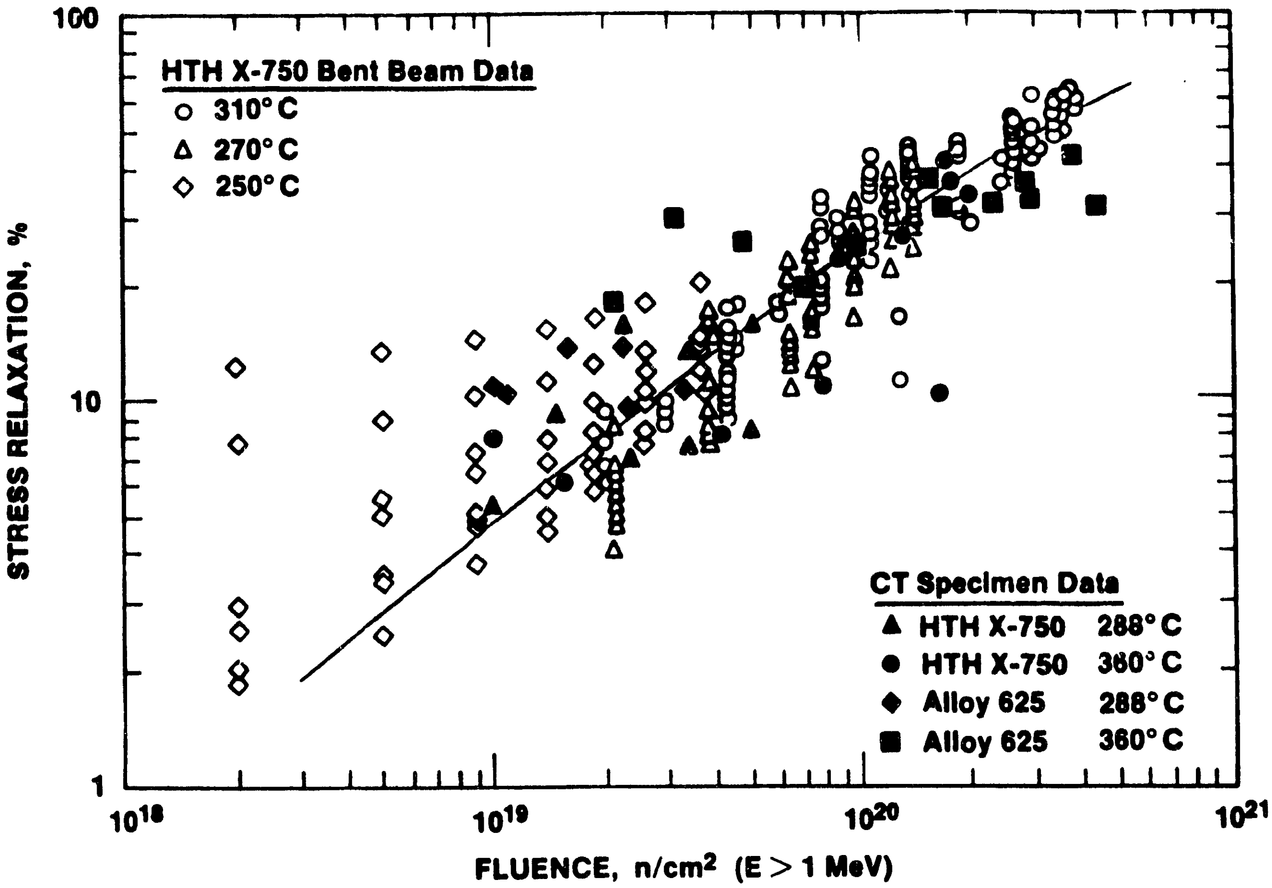
Irradiation damage function was assumed to be linear between 1 x 10^{18} and 2 x 10^{20} n/cm^2.

$$\textit{Irradiation Damage Factor} = 1.56\,F + 1.84$$

where F = fluence (in units of 10^{19} n/cm^2)

IN-REACTOR STRESS RELAXATION

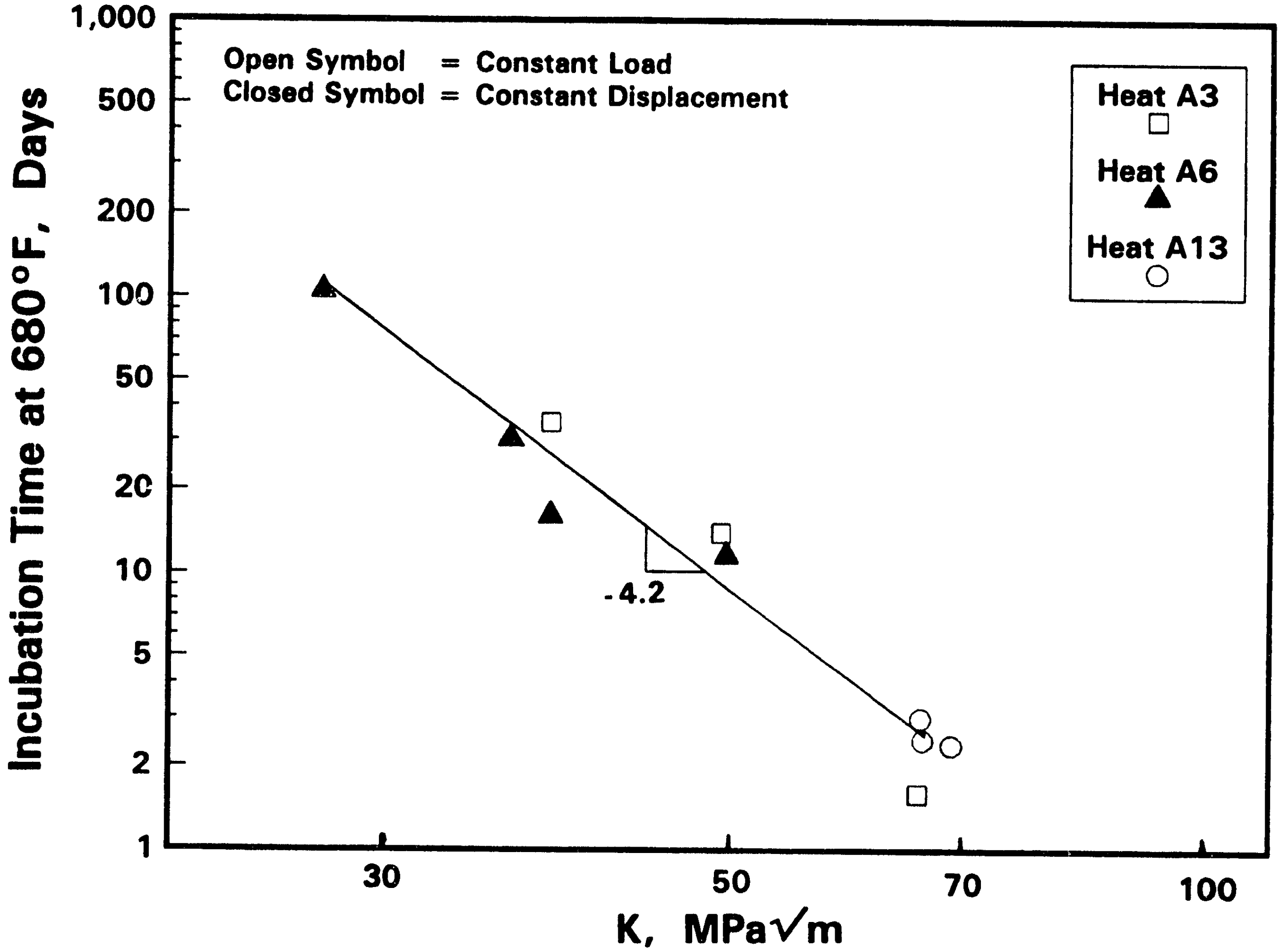
$$SR\% = 100\left[1 - 10^{-0.0208\,F^{0.78}}\right]$$



$$\frac{K}{Ko} = 10^{-0.0208\,F^{0.78}}$$

K_I DEPENDENCY OF SCC INCUBATION TIME FOR UNIRRADIATED HTH ALLOY X-750
(Based on Autoclave Tests)

$$t_{INC} = A\,K^{-4.2}$$



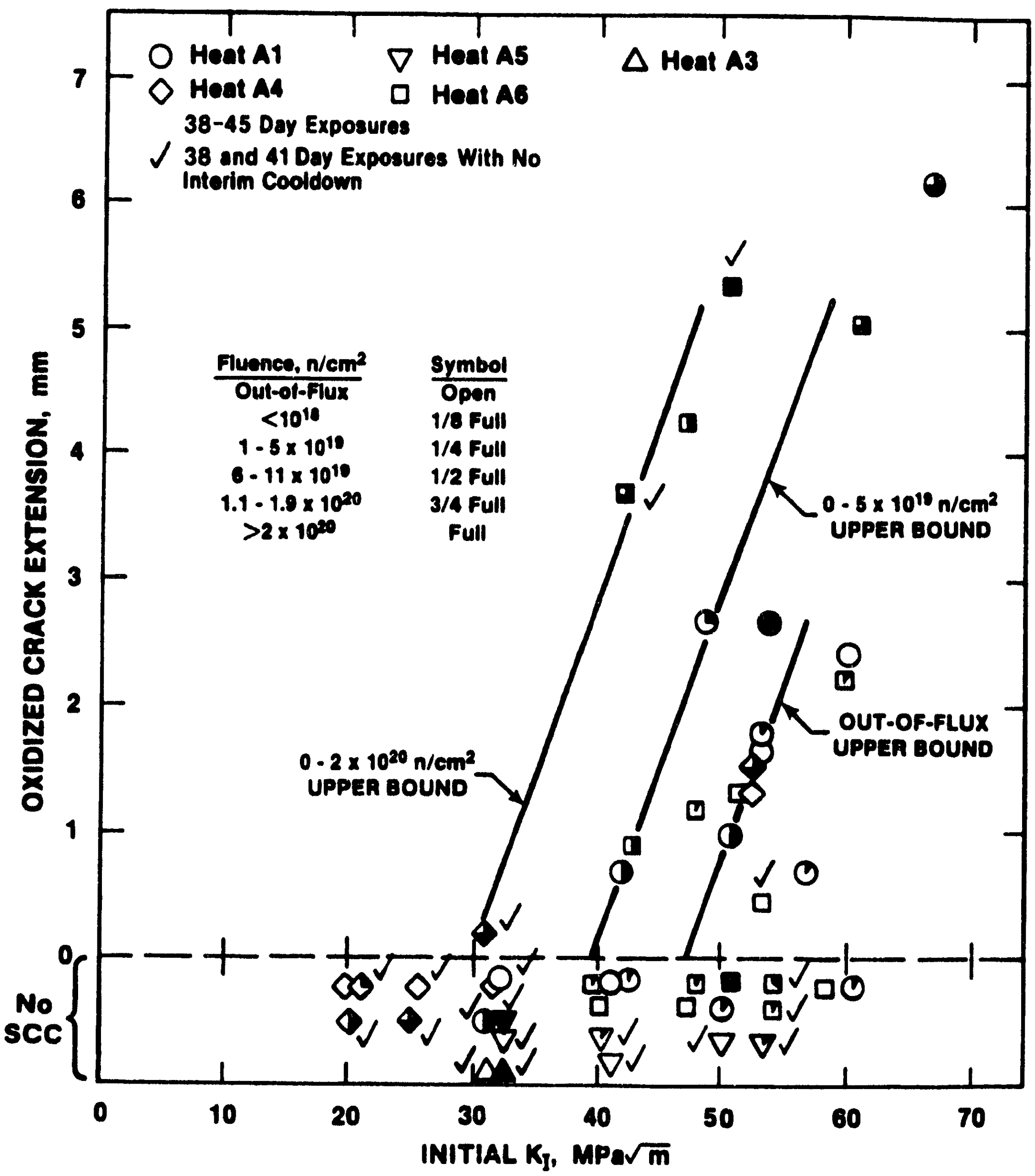
Data for Heat A3 were presented by KAPL at this Working Group Meeting.
The data point for Heat A6 at 27 MPa√m was supplied by D. Symons of Bettis.

SCC INCUBATION TIME FOR OUT-OF-FLUX & IN-REACTOR HTH ALLOY X-750

Unirradiated Condition: t_{INC} = 45 days; K = 47 MPa$\sqrt{m}$
(No relaxation or irradiation effects)

$$t_{INC}(days) = 4.74 \times 10^{8}\ K^{-4.2}$$

Irradiated Condition: t_{INC} = 38-45 days; K = 30 MPa$\sqrt{m}$



USAGE MODEL FOR PREDICTING IASCC INCUBATION TIME

$$t_{INC}(days) = 4.74 \times 10^{8}\ K^{-4.2} \qquad \text{(out-of-flux)}$$

$$\frac{K}{K_O} = 10^{-0.0208\ F^{0.78}}$$

$$\textit{Irradiation Damage Factor} = 1.56\,F + 1.84$$

Relative Usage Rate (RUR):

$$RUR = (1.56\,F + 1.84)\left(\frac{K}{K_O}\right)^{-4.2}$$

Incremental Usage (U):

$$\Delta U = \frac{(RUR)\ \Delta t}{t_{INC\ (@K_O)}} = 2.11 \times 10^{-9}\ (1.56\,F + 1.84)\ (10^{-0.0874\ F^{0.78}})\ K_O^{4.2}\ \Delta t$$

PREDICTED & MEASURED IN-REACTOR SCC INCUBATION TIMES
K = 30 MPa$\sqrt{m}$; F = 20 (20 x 10^{19} n/cm^2)

$t_{INC\text{-}MEASURED}$ = 38-45 days

$t_{INC\text{-}PREDICTED}$ > 45 days (Usage after 38-45 days was 0.55-0.62)

CONCLUSION

Even with the assumption of a severe irradiation damage effect based on postirradiation results, SCC should not occur during these in-reactor tests since usage was only 0.62 after 45 days. This suggests that SCC is accelerated by radiolysis effects and in-reactor creep processes which increase crack tip strain rates.

Thus, irradiation damage during in-reactor SCC tests performed under high flux conditions may be more severe than that associated with postirradiation tests because the latter does not account for radiolysis effects and in-reactor creep processes.

POSSIBLE IASCC MECHANISMS

Radiation Induced Segregation (RIS)

Nonequilibrium phenomenon causing segregation of trace and alloying elements to grain boundaries and other sinks and depletion of other elements.

Radiation Induced Microstructural Changes

Radiation can cause phase instabilities including precipitation of equilibrium and nonequilibrium phases.

Radiation can induce defect damage including cavities, loops and dislocations.

Precipitation and other microstructural features can affect SCC behavior by influencing mechanical and electrochemical properties.

Boron Transmutation Effects

Transmutation products, including helium and lithium, can reduce the cohesive strength of grain boundaries.

Radiation Effects on Deformation Characteristics

Radiation-induced displacement damage can alter strength and ductility, as well as promote planar slip, which adversely affects SCC resistance.

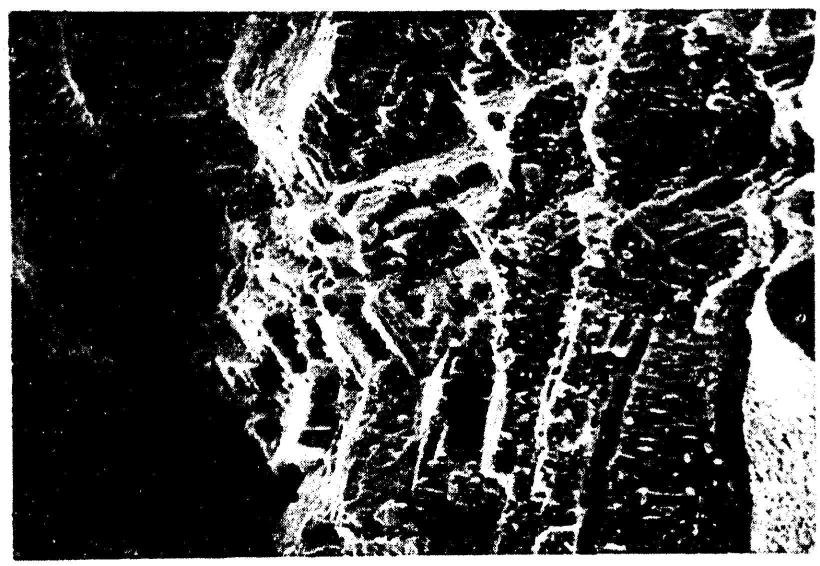
In-reactor creep effects can also influence SCC resistance.

Radiolysis Effects

Radiolysis of the water can significantly influence the electrochemical aspects of SCC.

Typical Intergranular Stress Corrosion Cracking and Evidence of Channel Fracture

20 μm



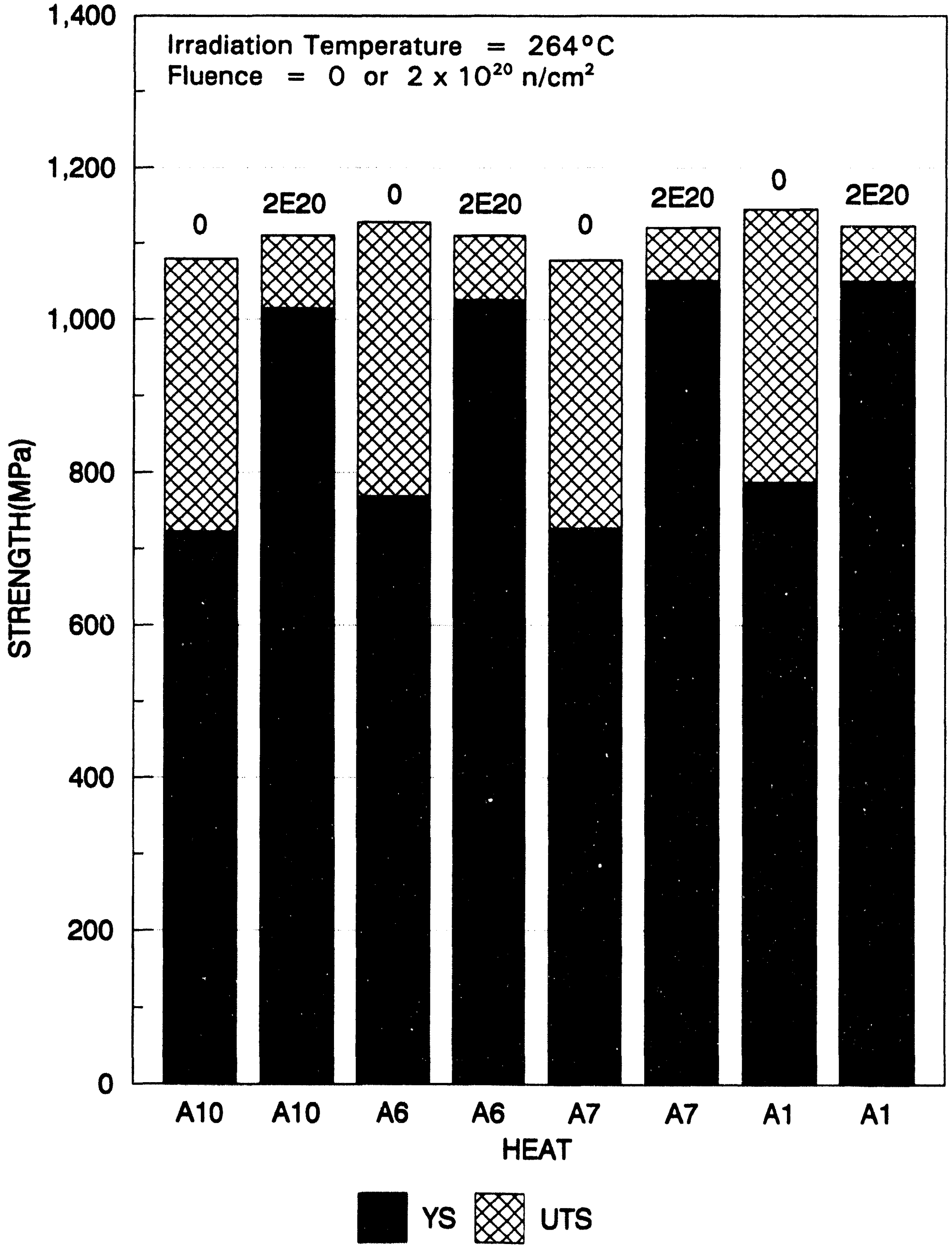
20 μm

HTH Alloy X-750 Heat A2

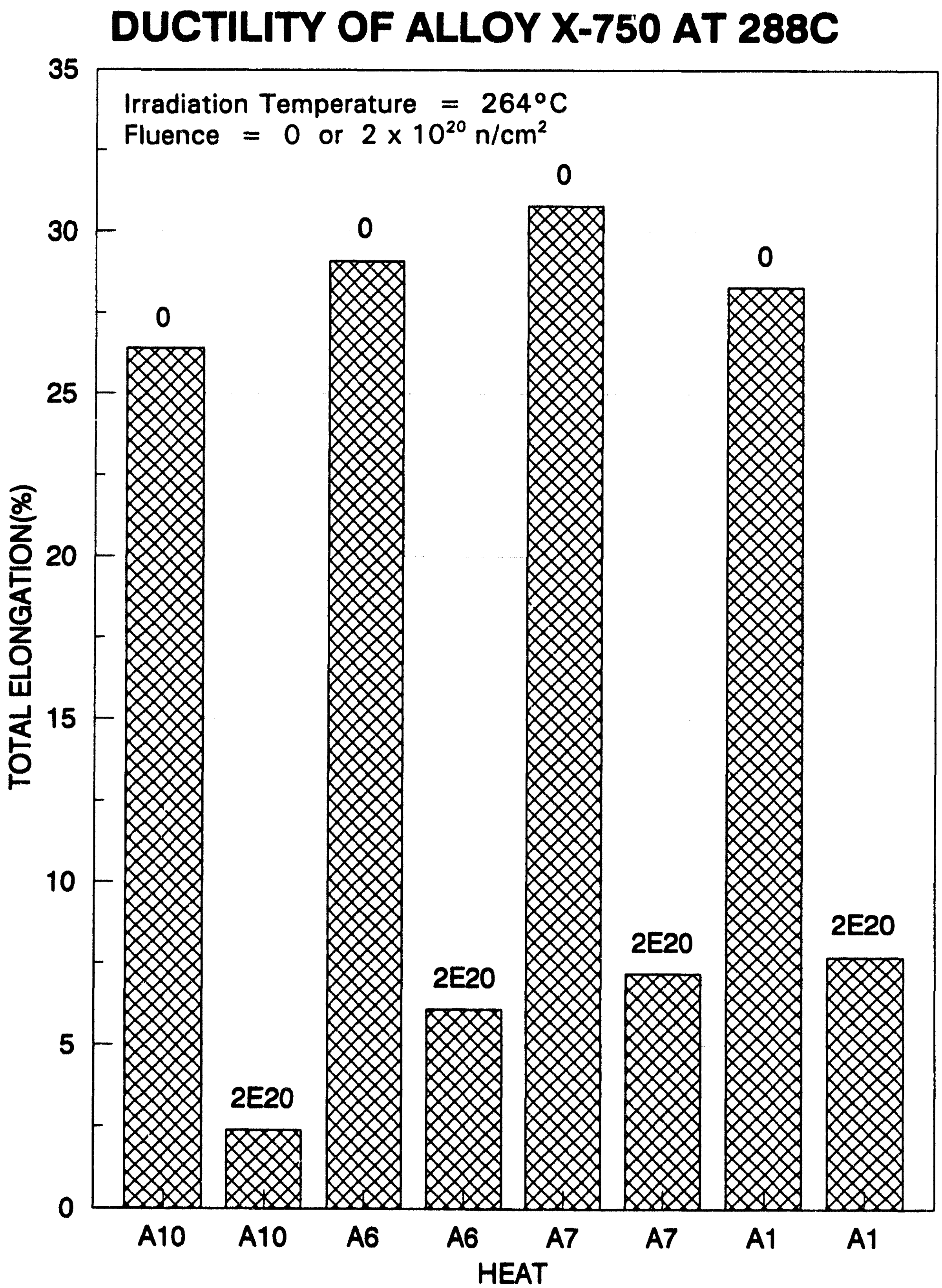
Fluence = 2×10^{19} n/cm^2 (E > 1 MeV)

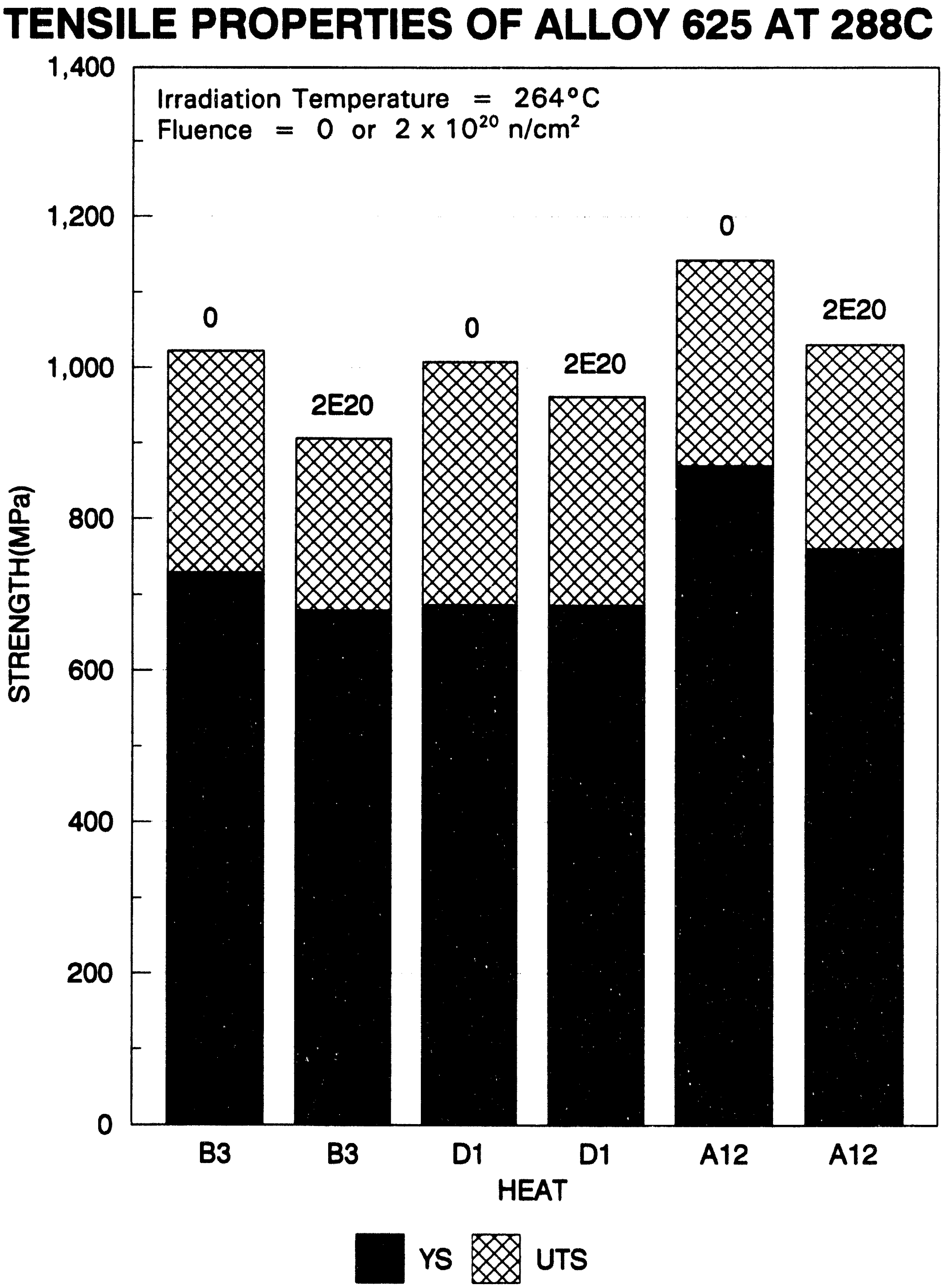
K_I = 55 MPa$\sqrt{m}$

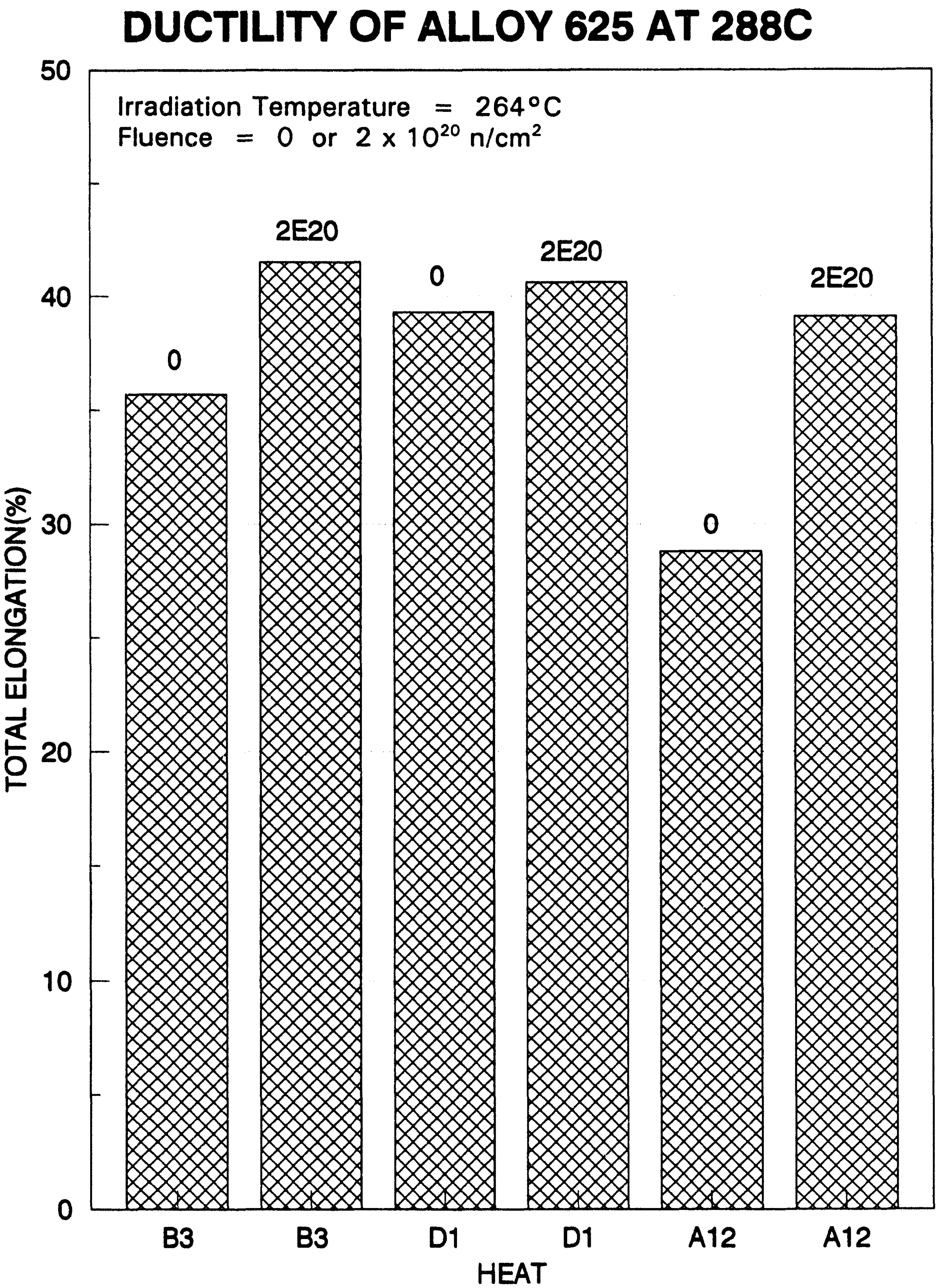
TENSILE PROPERTIES OF ALLOY X-750 AT 288C

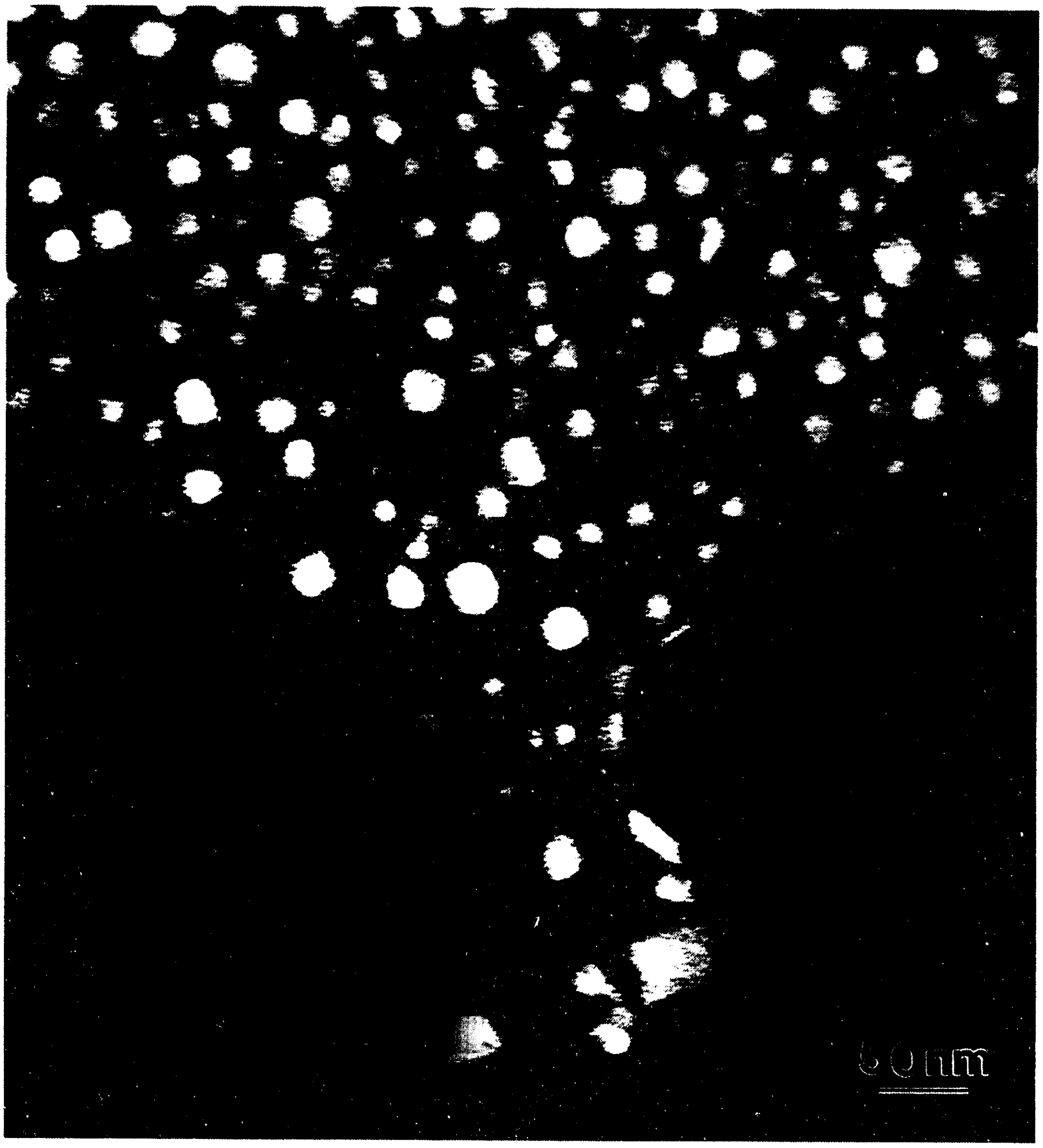


Fluence (E > 1 MeV) is given above each bar.

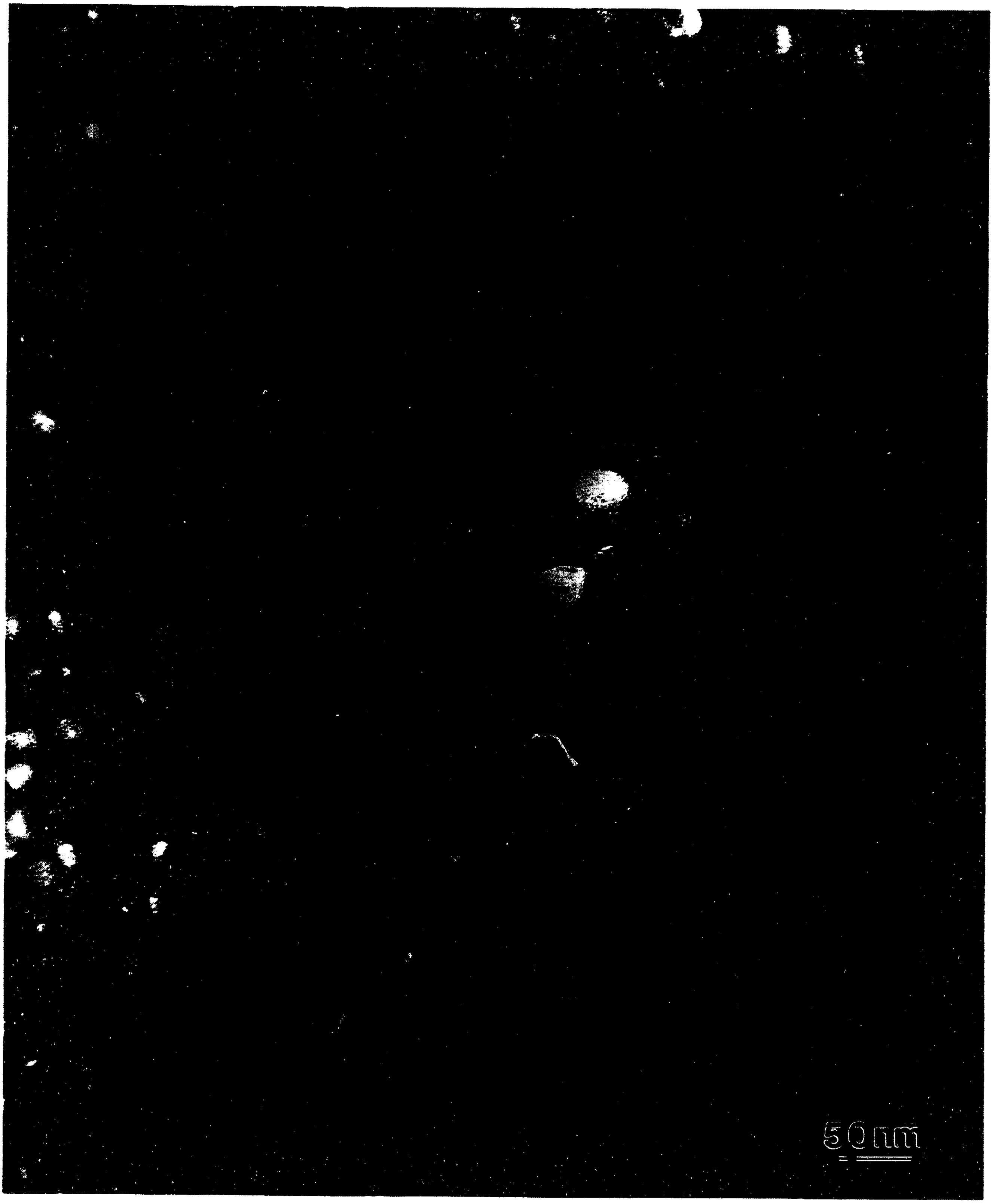

DUCTILITY OF ALLOY X-750 AT 288C
Irradiation Temperature = 264°C
Fluence = 0 or 2 x 10^20 n/cm^2
TOTAL ELONGATION(%)
35
30
25
20
15
10
5
0
0
2E20
0
2E20
0
2E20
0
2E20
A10
A10
A6
A6
A7
A7
A1
A1
HEAT


TENSILE PROPERTIES OF ALLOY 625 AT 288C
Irradiation Temperature = 264°C
Fluence = 0 or 2 x 10^20 n/cm^2
1,400
1,200
1,000
800
600
400
200
0
STRENGTH(MPa)
0
2E20
0
2E20
0
2E20
B3
B3
D1
D1
A12
A12
HEAT
YS
UTS

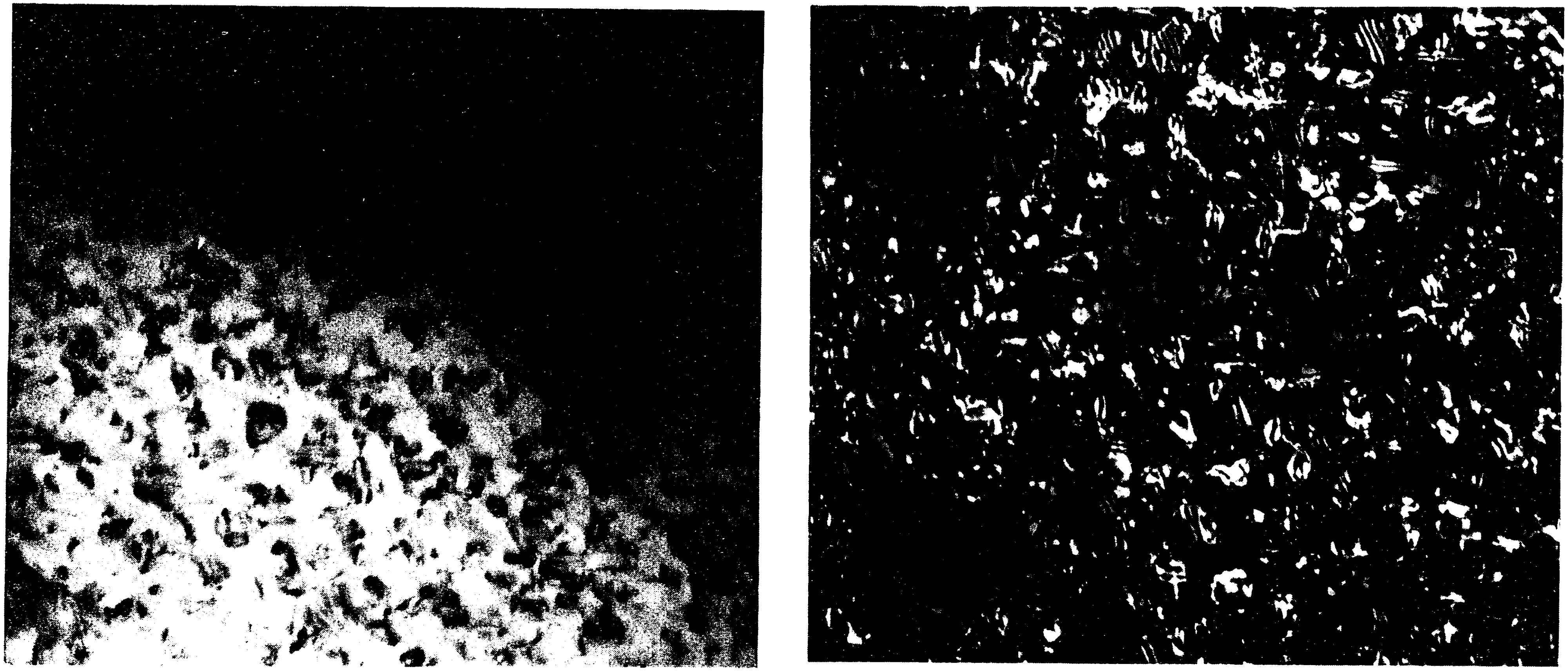

DUCTILITY OF ALLOY 625 AT 288C
Irradiation Temperature = 264°C
Fluence = 0 or 2 x 10^20 n/cm^2
0
2E20
0
2E20
0
2E20
50
40
30
20
10
0
TOTAL ELONGATION(%)
B3
B3
D1
D1
A12
A12
HEAT



[100] γ' dark field of unirradiated HTH Alloy X-750 (Heat A1) showing γ' precipitates along incoherent interface between grain boundary and $M_{23}C_6$.

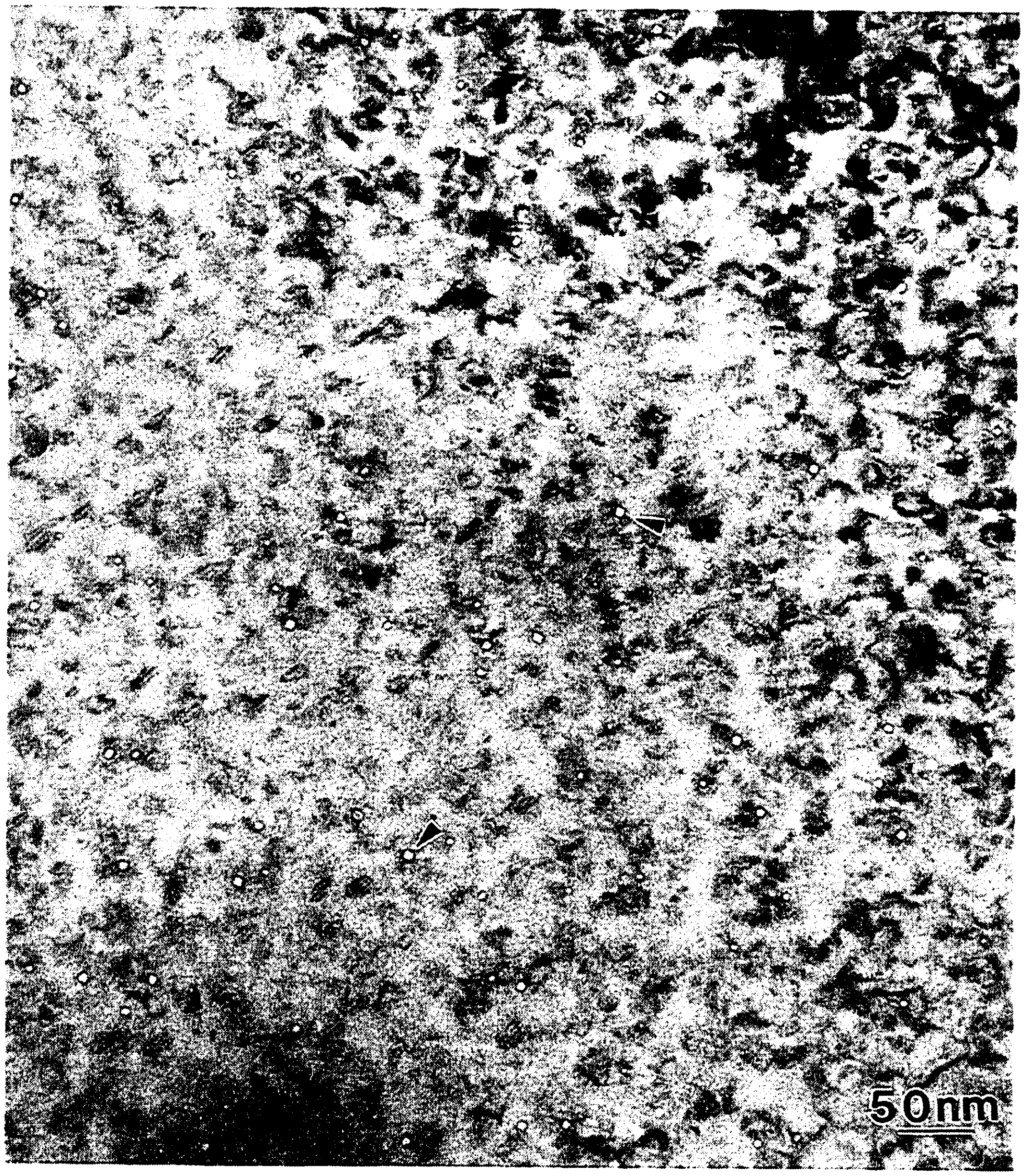


[100] γ' dark field of unirradiated HTH Alloy X-750 (Heat A1) showing large γ' precipitates and γ' film along grain boundary/$M_{23}C_6$ interface.

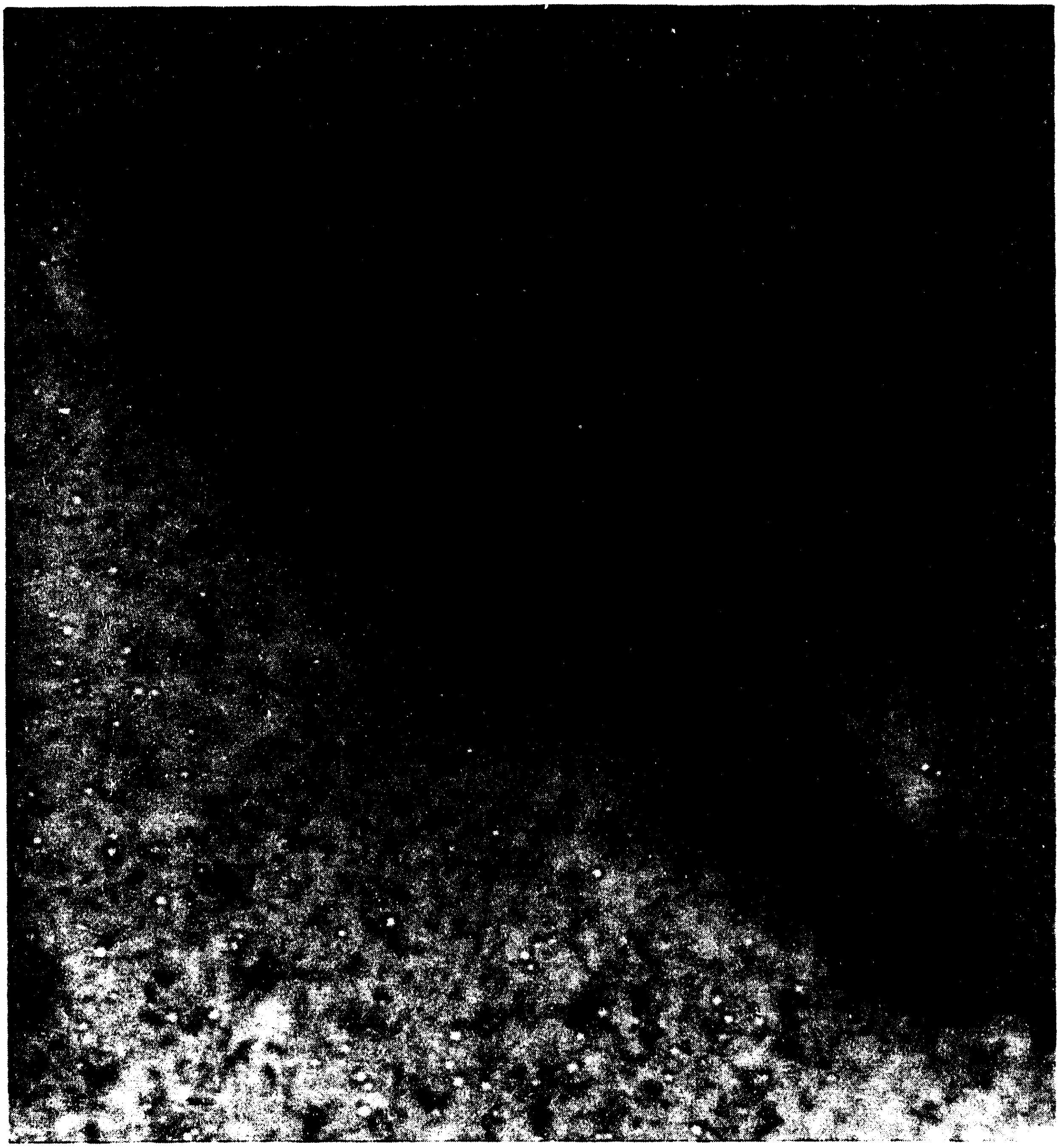


Bright field and dark field of HTH Alloy X-750 (Heat A1) irradiated to 2×10^{20} n/cm^2 showing faulted loops.

[100] γ' dark field of HTH Alloy X-750 (Heat A1) irradiated to 2×10^{20} n/cm^2 showing no change in γ' morphology. Dark bands inside γ' indicate presence of faulted loops.



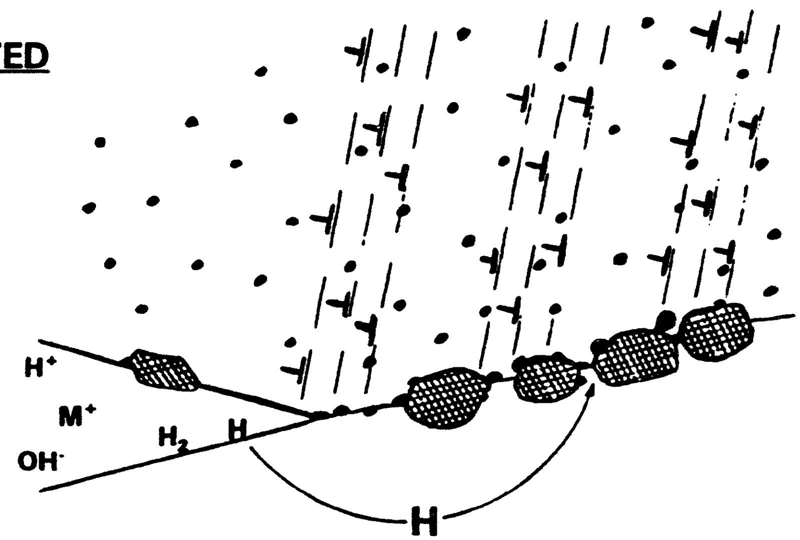
Bright field of HTH Alloy X-750 (Heat A1) irradiated to 2×10^{20} n/cm^2 showing cavities and faulted loops.



Bright field of HTH Alloy X-750 (Heat A1) irradiated to 2×10^{20} n/cm^2 showing cavities in matrix and intergranular $M_{23}C_6$. Note that cavities are not associated with grain boundary or precipitates.

SCC MECHANISMS IN HTH ALLOY X-750

UNIRRADIATED

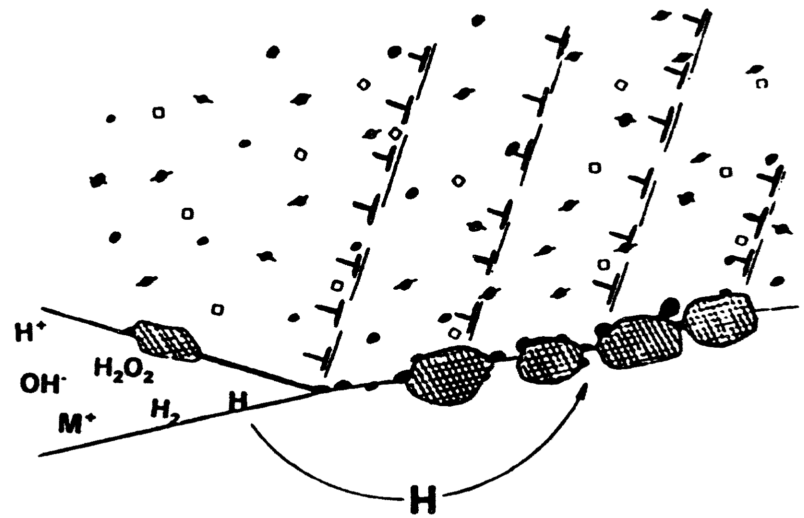


S and P segregation to G.B.
- Enhances effectiveness of hydrogen
- Lowers cohesive strength

Some evidence of γ' on G.B.
- γ' is highly anodic
- γ' serves as hydrogen trap

Beneficial effect of B at G.B.

IRRADIATED



High strength matrix increases peak σ_H
- Irradiation increases σ_{YS} by ~40%
- Strengthening due to cavities & loops

Strain localization concentrates strain at G.B.
- Dislocation channeling was operative (C.F.)
- Irradiation decreases σ_{UTS}/σ_{YS} from 1.5 to 1.1

In-Reactor Creep
- Enhances stress relaxation
- Enhances dislocation activity inside channels

Transmutation Effects [eg, B(n,α)]

Radiolysis Effects (eg, H_2O_2)

Radiation Induced Segregation?

SUMMARY

High irradiation levels significantly reduce SCC resistance in HTH X-750.

Heat-to-heat differences in IASCC correlated with differences in B content. Heats with less than 10 ppm B showed little or no effect of irradiation on HTSCC and LTCP.

Preliminary SCC usage model indicates that in-reactor creep processes, which relax stresses but also increase crack tip strain rates, and radiolysis effects accelerate SCC. Hence, irradiation damage during in-reactor SCC tests performed under high flux conditions may be more severe than that associated with postirradiation tests because the latter does not account for radiolysis and in-reactor creep effects.

Preliminary mechanism studies indicate that irradiation of HTH X-750 significantly increases yield strength, decreases ductility, enhances planar slip (based on evidence of channel fracture), and induces cavities and faulted loops.

Absence of in-reactor SCC and excellent LTCP resistance for Alloy 625 demonstrates its superiority to HTH X-750 for PWR applications.

Irradiation to 2 x 10^{20} n/cm^2 has very little effect on strength or ductility of Alloy 625.

DATE
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7/6/94

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