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W. J. Mills, M. R. Lebo and J. J. Kearns

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INHIBITION OF STRESS CORROSION CRACKING OF ALLOY X-750 BY PRESTRAIN

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ABSTRACT

Tests of precracked and as-notched compact tension specimens were conducted in 360°C hydrogenated water to determine the effect of prestrain on the stress corrosion cracking (SCC) resistance of Alloy X-750 in the HTH, AH and HOA heat treated conditions. Prestraining is defined as the intentional application of an initial load (or strain) that is higher than the final test load. Prestrain was varied from 10% to 40% (i.e., the initial to final load ratios ranged from 1.1 to 1.4). Other variables included notch root radius, stress level and irradiation. Specimens were bolt-loaded to maintain essentially constant displacement conditions during the course of the test. The frequent heat up and cooldown cycles that were necessary for periodic inspections provided an opportunity to evaluate the effect of test variables on rapid low temperature crack propagation to which this alloy is subject.

For Condition HTH, application of 20% to 40% prestrain either eliminates or significantly retards SCC initiation in as-notched specimens and the onset of crack growth in precracked specimens. In addition, this procedure reduces the propensity for low temperature crack growth during cooldown. Similar results were observed for precracked HOA specimens. Application of 20% prestrain also retards SCC in as-notched and precracked AH specimens, but the effects are not as great as in Condition HTH. Prestraining at the 10% level was found to produce an inconsistent benefit.

In-reactor SCC testing shows that prestrain greatly improves the in-flux and out-of-flux SCC resistance of Condition HTH material. No SCC was observed in precracked specimens prestrained 30%, whereas extensive cracking was observed in their nonprestrain counterparts.

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INTRODUCTION

Alloy X-750 is used in high strength fastener applications in light water reactors because of its high temperature strength and its resistance to corrosion and load relaxation. However, this material is susceptible to intergranular stress corrosion crack initiation and growth at temperatures above 250°C. In addition, rapid low temperature crack propagation (LTCP) can occur at temperatures below 150°C⁽¹⁻⁴⁾ when a crack or crack-like defect is present and the applied stress intensity factor is above a critical value. Because LTCP rates are extremely high (on the order of 5 mm per minute), final specimen separation occurs in a short period of time once low temperature cracking commences. While the critical K_I level for LTCP ($K_{ISCC-LT}$) is substantially higher than the threshold level for high temperature stress corrosion cracking (SCC), it can be considerably less than half of the fracture toughness. Evidence of SCC in Alloy X-750 components has been observed in light water reactors.⁽⁵⁻⁷⁾

The two most common heat treatments for Alloy X-750 fasteners are termed Conditions AH and HTH. The original fastener materials in the Condition AH treatment (stress equalized at 885°C for 24 hours and aged at 704°C for 20 hours) were susceptible to high temperature SCC and had variable resistance to LTCP. The Condition HTH treatment (solution annealed at 1093°C for 1 to 2 hours and aged at 704°C for 20 hours) improved SCC resistance at both low and high temperatures. In addition, a newer treatment termed Condition HOA (solution annealed at 1121°C for 0.5 hour and aged at 760°C for 96 hours) provides an overaged matrix designed to further improve SCC performance, but at decreased strength levels.

In this paper the concept of prestrain is examined as a means of SCC mitigation for Alloy X-750 in the AH, HTH and HOA heat treated conditions. Prestrain is the intentional application of an initial load (displacement) that is higher than the final load (displacement) in order to reduce the local elastic-plastic stress at a notch root or crack tip. Because SCC is sensitive to the stress state immediately ahead of a machined notch or crack tip, reduction of these stresses by prestrain is expected to enhance SCC resistance. Prestrain effects were evaluated by autoclave testing of as-notched and precracked compact tension (CT) specimens that were prestrained 0% to 40% before being exposed to 360°C water. Specimens were periodically cooled in high hydrogen water which enabled characterization of prestrain effects on LTCP. In-reactor SCC testing was also performed to determine if prestrain benefits are maintained at fluences up to 2×10^{20} n/cm² ($E > 1$ MeV).

CONCEPT OF PRESTRAIN-INDUCED STRESS BENEFIT

Load-Partial Unload Case in Notched Geometry

The effect of partial unloading on the stress and strain conditions at a notch surface and net section is shown in Figure 1. In this example, two identical notched samples, both with a theoretical stress concentration factor (K_t) of 6, are loaded to a net section stress of 400 MPa. The first sample is direct loaded to 400 MPa (see A on Figure 1), whereas the second is loaded to 480 MPa (B) and then unloaded to 400 MPa (A), representing a 20% prestrain. As portrayed in Figure 1, direct loading to 400 MPa produces a strain of 0.22% averaged across the net section, and a surface strain of approximately $6 \times 0.22\% = 1.32\%$, as a result of the strain concentration effect (i.e., because the surface elastic stress at the notch root is well beyond the yield strength, K_t is more a measure of strain concentration than the actual stress concentration). Therefore, the surface stress is 940 MPa for direct loading (C). When the second specimen is loaded to 480 MPa, the average strain is 0.26% and the surface strain is approximately $6 \times 0.26\% = 1.56\%$. As shown in Figure 1, the corresponding surface stress is 950 MPa (D). Reduction in the net section stress to 400 MPa reduces the average strain to 0.22% and the surface strain to 1.32%. The corresponding surface stress is now 490 MPa (E) due to elastic unloading. Thus, in this formulation, prestraining reduces the calculated surface stress by nearly 50% (i.e., 940 MPa for direct loading versus 490 MPa for loading and partial unloading). As may be observed from the construction in Figure 1, the final local stress at the notch decreases as the percent prestrain increases and as the stress concentration increases. Each of these factors broadens the band depicting the (elastic) unloading strain while the maximum stress during prestrain application increases slightly.

Load-Partial Unload Case in Precracked Geometry

The effect of partial unloading on the stress state ahead of a crack is illustrated in Figure 2. Under direct loading conditions, the maximum principal stress, σ_Y , ahead of a crack is given by the linear-elastic stress field equation,

$$\sigma_Y = \frac{K_I}{\sqrt{2\pi r}} \quad (1)$$

where K_I is the applied stress intensity factor and r is the distance directly ahead of the crack tip. For simplicity, it is assumed that the actual stress is truncated at the yield strength (σ_{YS}) level to account for plasticity effects. The overall stress profile ahead of a crack under direct

loading is then given by the solid line in Figure 2(a). The key to understanding prestrain effects is to recognize that during partial unloading the material directly ahead of a crack tip behaves in a linear-elastic manner. Accordingly, the degree of stress reduction associated with a partial unloading, represented in Figure 2(b), is computed from Equation 1. The vertical distance represented by the arrows in Figure 2(b) shows the magnitude of the stress drop both inside and outside the plastic zone during the unloading. To obtain the final stress state after elastic unloading, the elastic-plastic stress state in Figure 2(a) is reduced by the magnitude of the arrows in Figure 2(b) to produce the stress profile represented by the tips of the arrows in Figure 2(c). It is seen that the partial unloading dramatically reduces the crack tip stress field relative to that for direct loading. This is a key effect because the improved elastic-plastic stress distribution occurs in the critical zone close to the metal-water interface where SCC mechanisms are active. Increasing the percent prestrain produces a greater reduction in local stresses which is expected to further retard cracking.

EXPERIMENTAL PROCEDURE

The chemical compositions of the test materials are provided in Reference (1). The heat numbers used herein (AH Heat D8, HTH and HOA Heat A6, and HTH Heat B8) are consistent with those in Reference (1).

Tests were performed on bolt-loaded CT specimens with a width of 20.3 mm and a thickness of 10.2 mm. Those tested in the as-notched condition had a notch depth of 10.2 mm and root radii of 0.06, 0.25 and 0.76 mm; corresponding K_t values were 9.7, 4.9 and 2.8, respectively. Notches were finished by low-stress grinding. Precracked specimens were fatigue precracked to an a/W of 0.5 at a maximum K_I level of 20 MPa \sqrt{m} .

All specimens were bolt-loaded to the desired notch mouth opening displacement (NMOD) by the direct continuous torquing of the bolt. The corresponding load was determined by developing load versus NMOD calibration curves for identical notched or precracked specimens. To determine K_I levels at 360°C, constant displacement conditions were assumed on heating, which is a good assumption for Alloy X-750 due to its excellent stress relaxation resistance, and calculated load levels used in the K_I solution⁽⁸⁾ were adjusted by the ratio of elastic moduli at room temperature and 360°C (i.e., a factor of 1.13).

To introduce prestrain, specimens were torqued to the desired prestrain NMOD level and then partially unloaded to the final NMOD level. Percent prestrain is defined by $100 \times (\text{initial NMOD} - \text{final NMOD}) \div \text{final NMOD}$. For precracked specimens, which were loaded in the linear-elastic domain, the percent overload in terms of K_I is equivalent to the percent prestrain.

Tests were performed in 360°C hydrogenated water with a room temperature pH between 10.1 and 10.3. During the first 70 days of exposure, the hydrogen level at temperature was about 35 cc H₂/kg H₂O and during cooldown it was controlled between 40 and 60 cc H₂/kg H₂O to give specimens an increased opportunity to undergo LTCP. Beyond this period, hydrogen levels were between 40 and 60 cc H₂/kg H₂O at temperature and reduced below 20 cc H₂/kg H₂O during cooldown to prevent low temperature cracking in new specimens added to the test.

Tests were periodically interrupted to allow specimen examinations. Inspection of as-notched specimens consisted of a 60X stereo exam of the notch root for crack initiation. The faces of each precracked specimen were examined for surface crack extension. Residual bolt-load measurements were made on selected precracked specimens to detect crack tunnelling and

obtain reliable crack growth data in specimens with no visible surface crack extension. This procedure was performed without substantial unloading of the specimen, so subsequent SCC behavior was unaffected. In this procedure, the bolt load was transferred to a tensile machine, while never unloading the crack or notch tip more than 2% to 3%. The crack length was then determined from the residual bolt load via the Saxena-Hudak compliance relationship.⁽⁹⁾ This process was used in reverse to reload specimens for further exposure. When desired exposure times were reached, specimens were destructively examined to measure the amount of crack extension visually.

Prestrained and nonprestrained bolt-loaded specimens were irradiated at approximately 360°C to fluences between 1.9×10^{19} and 4×10^{20} n/cm² ($E > 1$ MeV). Control specimens were also included in the same reactor loop, but in out-of-flux positions. During high temperature exposure, the water contained 40 to 60 cc H₂/kg H₂O. Hydrogen levels were lowered to less than 20 cc H₂/kg H₂O during cooldown to minimize the potential for LTCP. After irradiation, specimens were broken apart to measure crack extension. The extent of LTCP was determined by combining light microscopy and scanning electron microscopy observations to detect any intergranular SCC extending beyond the dark intergranular region.

RESULTS AND DISCUSSION

Effect of Prestrain on Notched Specimens

The effects of 10% to 30% prestrain on crack initiation times in Condition AH specimens with blunt ($\rho = 0.76$ mm) and sharp ($\rho = 0.06$ mm) notches are illustrated in Figure 3. At intermediate and high net section stress levels, SCC is moderately retarded by 20% prestrain, with initiation times being increased by a factor of two for both notch radii. At the lowest net section stress levels, 20% prestrain effects are more pronounced. In the sharply notched specimen prestrained 20%, no SCC was observed after 161 days, whereas its nonprestrained counterpart cracked after just 18 days. For the blunt-notch specimen with a net section stress of 380 MPa, 20% prestrain produced more than tripled SCC initiation times. A 30% prestrain at this baseline stress level is seen to produce a factor of five increase in cracking time, indicating that greater prestrain levels further enhance retardation. Application of prestrain in the low stress regime is believed to reduce notch-tip stresses to near-threshold levels, so prestrain effects are enhanced. Figure 3 also shows that 10% prestrain has no effect on SCC behavior for Condition AH.

Prestrain effects on Condition HTH material were evaluated by testing bolt-loaded specimens with notch root radii of 0.13, 0.25 and 0.76 mm. Specimens loaded to PSES levels ranging from 2030 to 8330 MPa were exposed to 360°C water for 273 weeks. The prestrain levels were 0%, 10%, 20% and 30%. A plot of the cumulative frequency of SCC initiation with exposure time is shown in Figure 4. The data were separated based on the applied peak surface elastic stress (PSES), which is defined as the product of K_t and nominal stress. This parameter is effective in normalizing data for various root radii.⁽¹⁾ For the nonprestrained condition, all specimens with PSES levels between 5920 and 8330 MPa cracked within 147 weeks, all specimens with PSES values between 4530 and 5070 MPa cracked after 237 weeks, and 57% of the specimens with PSES values between 2030 and 3550 MPa cracked after 273 weeks. Analysis of these data in Reference (1) revealed a power-law relationship between SCC initiation time and PSES with a stress exponent of -1.4.

The efficacy of prestrain on HTH specimens is also demonstrated in Figure 4. Prestrain levels of 20% and 30% are effective in mitigating SCC as these specimens were immune to cracking through 273 weeks, regardless of applied PSES level. Although it is not possible to quantify the benefit of 20% to 30% prestrain because no initiation occurred in any specimens

prestrained to this level, it is noted that survival times in the highest PSES regime are 8 times longer than the initiation time for the first nonprestrained specimens to crack.

While 10% prestrain is less effective than 20% and 30% prestrain, it did confer some protection. In general, at any given exposure time, the cumulative frequency of initiation in the 10% prestrain specimens is about half that in nonprestrained specimens.

Comparison of the SCC responses in Figures 3 and 4 shows that Condition HTH exhibits a much greater prestrain benefit than Condition AH, but the reason for the different responses is not fully understood. The data suggest that the stress dependency of Condition HTH should be much greater than that for Condition AH. However, as-notched specimen data analyzed in Reference (1) show that the stress dependence for the two material conditions is not radically different, as n is -0.8 and -1.4 for the AH and HTH conditions, respectively. While the 75% greater stress exponent for Condition HTH accounts for a portion of its increased prestrain benefit, the markedly different prestrain responses for Conditions AH and HTH suggest that other factors are also playing a role. Specifically, it is postulated that the relatively small prestrain benefit for Condition AH may be related to its propensity for off-root cracking, as illustrated in Figure 5. In some specimens SCC initiates near the notch tip, but in others cracking initiates away from the notch tip where the applied stresses are substantially lower. This behavior occurs because the AH grain boundaries are highly susceptible to SCC, so cracking can occur in the lower stressed regions when local notch tip conditions are not conducive to cracking. Prestrain has a maximum benefit at the notch tip where the stress concentration is greatest. In AH material, however, the adjacent material is still susceptible to SCC so the overall benefit is rather small. Grain boundaries in Condition HTH heats are far more resistant to SCC, so cracks tend to be restricted to the notch tip region where prestrain is most effective. As a result, the prestrained-induced reduction in local stresses ahead of a notch has a much greater influence on SCC susceptibility for Condition HTH.

Effect of Prestrain on Precracked Specimens

The effect of prestrain on SCC behavior for precracked Condition AH specimens loaded to three different K_I levels is shown in Figure 6. Comparison of nonprestrained and 20% prestrained data shows a definite lengthening of the incubation period for prestrained specimens. Crack growth is in progress within 3 days in all six of the nonprestrained specimens. Prestrain retards crack incubation by different degrees, but more than 7 days are

needed before growth is underway in all six prestrained specimens. Hence, crack incubation times are doubled as a result of 20% prestrain. This modest effect reflects a small stress dependency on crack incubation behavior, as shown in Figure 6 where increasing the applied K_I from 24 to 43 MPa \sqrt{m} has little effect on crack incubation time.

Because prestrain influences the local stress state very near the crack tip, it has no effect on cracking behavior once the crack extends a small distance. This is shown in Figure 6 where crack growth rates, corresponding to the slope of the crack extension versus time curves, are essentially the same for nonprestrained and prestrained specimens.

Prestrain effects for Condition HTH Heats A6 and B8 are shown in Figures 7 and 8. The steep curve segments in these crack extension versus exposure time plots imply rapid LTCP during autoclave cooldown. Nonprestrained specimens are seen to exhibit high temperature crack growth at all three K_I levels and LTCP during autoclave cooldown at the two higher K_I levels. This is consistent with a $K_{ISCC-LT}$ value of about 45 MPa \sqrt{m} for Heat A6 in high hydrogen water.⁽¹⁾ Companion specimens from Heat A6 that were prestrained 20% reveal no LTCP at any of the K_I levels and sustained high temperature SCC in only one of the highest loaded specimens after 100 days. Similar behavior is displayed by Heat B8 except that LTCP did occur in one of two specimens at the highest K_I level. These findings demonstrate that 20% prestrain produces a strong beneficial effect in Condition HTH, although some cracking can still occur at very high K_I levels. The increased benefit over Condition AH reflects a high stress sensitivity on SCC incubation. Condition HTH incubation times are a power-law function of K_I to the -4.1 power,⁽¹⁾ indicating that SCC is sensitive to crack tip stress levels.

Results of tests at 10% prestrain are much less dramatic. While 10% prestrain protects HTH specimens against cracking at 33 MPa \sqrt{m} , results are mixed at 50 MPa \sqrt{m} and there is no significant protection at 66 MPa \sqrt{m} .

Application of a 20% prestrain also produces a strong beneficial effect on HOA Heat A6, as demonstrated by the data in Figure 9. While nonprestrained specimens are susceptible to low and high temperature cracking in less than 21 days of exposure in 360°C water, less than 0.2 mm of stable cracking is observed in their 20% prestrain counterparts after 224 days.

To quantify the prestrain benefit for Condition HTH, high temperature SCC incubation times for prestrained specimens are compared with data for nonprestrained specimens in Figure 10. The reference curve⁽¹⁾ is based on crack incubation times for five HTH heats tested under constant-displacement and constant-load conditions. Included in this data set are specimens

from Heats A6 and B9, the two heats included in the prestrain comparison. The effect of 20% to 40% prestrain in eliminating or retarding crack growth is shown by the open or partly open squares and triangles. The majority of prestrained specimens revealed no evidence of SCC at the time they were discontinued for destructive examination. In cases where some high temperature SCC was observed, the time for the start of crack growth was estimated by correcting the total exposure time by the amount of time required to grow the crack from 0.08 mm to the observed size. These corrections were based on the crack growth rate equation (Eq. 4) in Reference (1). In the specimens that did crack, prestrain delayed incubation by an order of magnitude at low and intermediate K_I levels and by much more at the highest levels. Moreover, most of the prestrained specimens showed no SCC after 537 days demonstrating that on average, prestrain benefits are even greater than a factor of ten in incubation time. There were isolated cases of highly stressed specimens failing by LTCP during cooldown, but the data clearly show that both high and low temperature protection is provided by 20% to 40% prestrain.

Effect of Irradiation on Prestrain Benefit

The in-reactor performance of Condition HTH specimens, summarized in Figure 11, shows the dramatic benefit of 30% prestrain in eliminating both high and low temperature SCC. Substantial irradiation-assisted SCC occurred in all in-flux control specimens; some of the cracking was due to high temperature SCC and some due to LTCP. The highest stressed out-of-flux control specimen also showed evidence of SCC. By contrast, no cracking occurred in companion prestrained specimens. This demonstrates that the beneficial effects of prestrain in preventing both modes of cracking are maintained in-reactor at fluences up to 2×10^{20} n/cm² ($E > 1$ MeV).

CONCLUSIONS

Tests of as-notched and precracked specimens were conducted in 360°C water to characterize the effectiveness of prestrain in retarding SCC in Conditions HTH, AH and HOA Alloy X-750. Specimens were intentionally cooled in high hydrogen water to evaluate the role of prestrain in inhibiting LTCP. The results of this program are summarized below.

1. For Condition AH specimens, prestrain at the 20% level has only a moderate effect in retarding crack initiation from a notch and crack incubation from a precrack. Prestrain has no effect on crack growth rates once the crack has propagated a small distance.
2. The stress dependence for SCC initiation and incubation in Condition HTH specimens is considerably greater than that for Condition AH specimens.
3. For Condition HTH specimens, prestrain at the 20% to 40% levels has a strong inhibiting effect on both SCC initiation in as-notched specimens and the onset of crack growth in precracked specimens. Protection is provided against both high temperature SCC and low temperature cracking. Prestrain at the 20% to 40% levels produces at least an order of magnitude improvement in high temperature SCC incubation times.
4. Application of 10% prestrain produces an inconsistent benefit for Condition HTH, causing a small benefit for as-notched specimens but little or no benefit for precracked specimens.
5. Prestrain at the 20% level produces a strong benefit for Condition HOA specimens.
6. The beneficial effects of prestrain in preventing both low and high temperature SCC are maintained in-reactor.

ACKNOWLEDGEMENT

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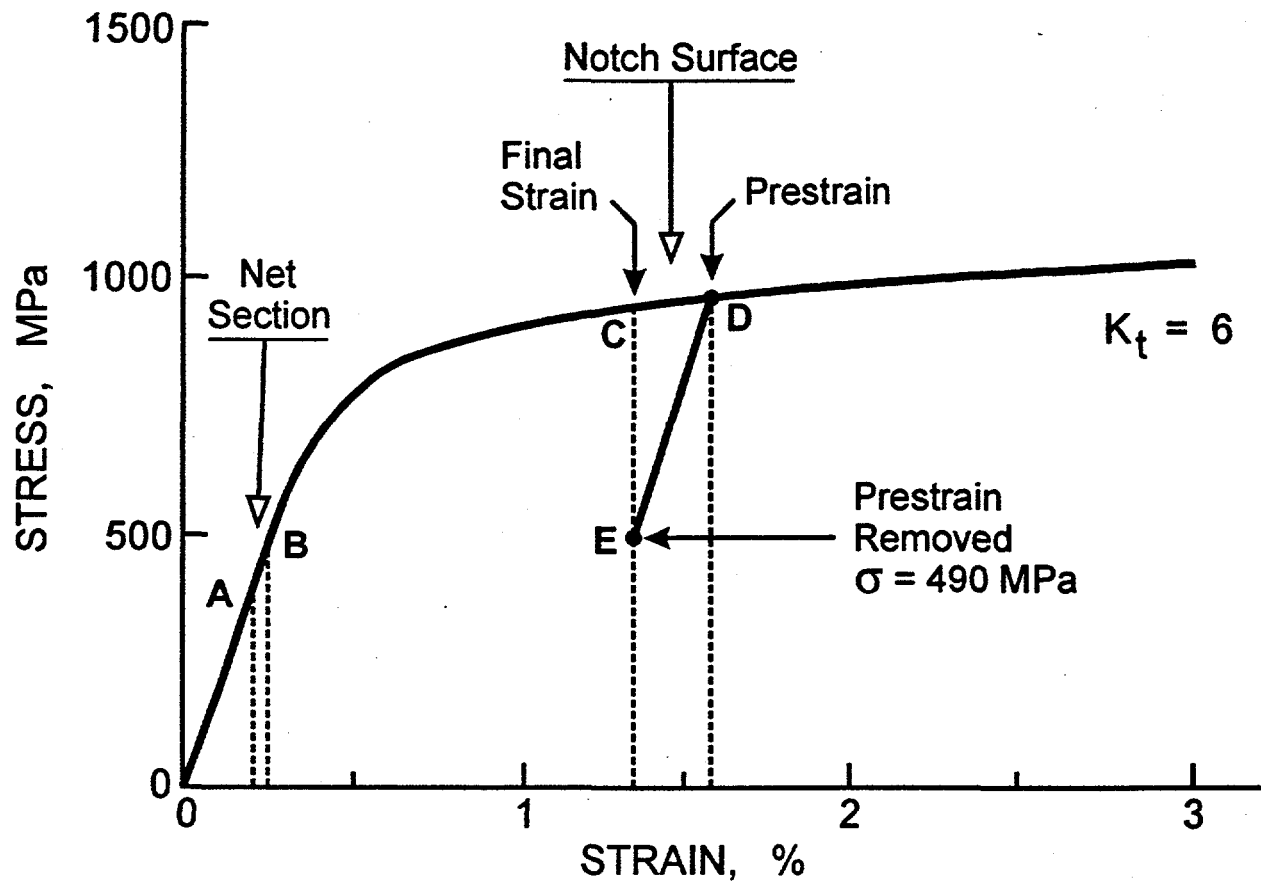


Figure 1. Conceptual analysis of effect of 20% prestrain on local stresses at the surface of a notch.

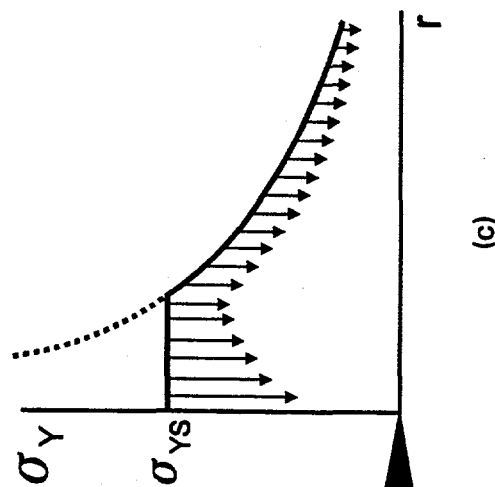
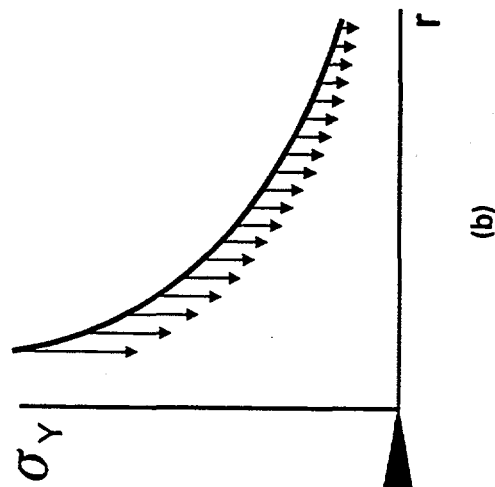
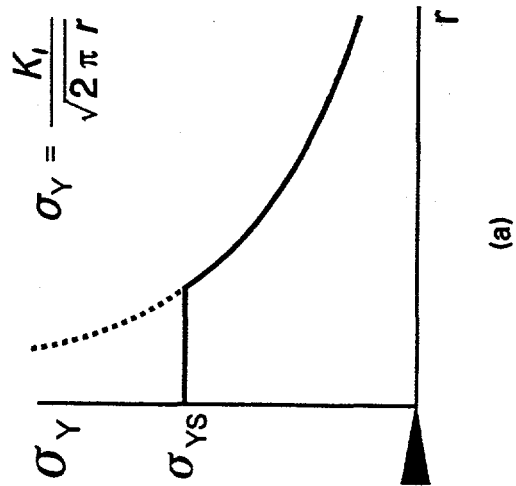


Figure 2. Conceptual analysis of prestrain effects on stress state ahead of a crack. (a) Maximum principal stress (σ_Y) given by the linear-elastic stress field equation is truncated at the yield strength to account for crack tip plasticity. (b) During partial unloading, material ahead of crack behaves in a linear-elastic manner, so all elastic stresses are reduced by the same percentage. (c) Stress state after partial unloading is obtained by combining the stresses in (a) with the decrease in stress in (b). Note dramatic reduction in elastic-plastic stresses directly ahead of crack.

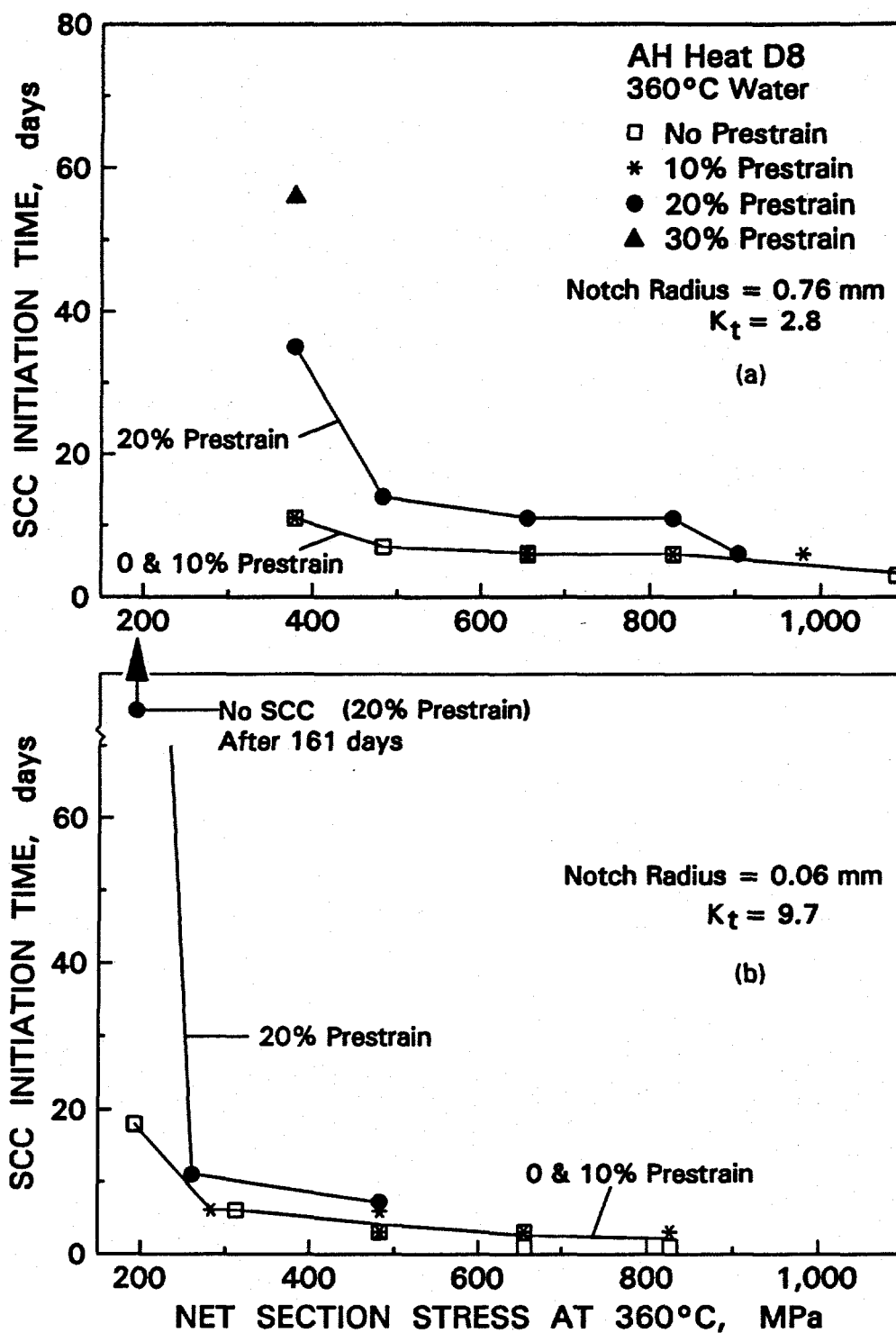


Figure 3. Effect of prestrain on SCC initiation in as-notched Condition AH specimens with root radii of (a) 0.76 mm and (b) 0.06 mm.

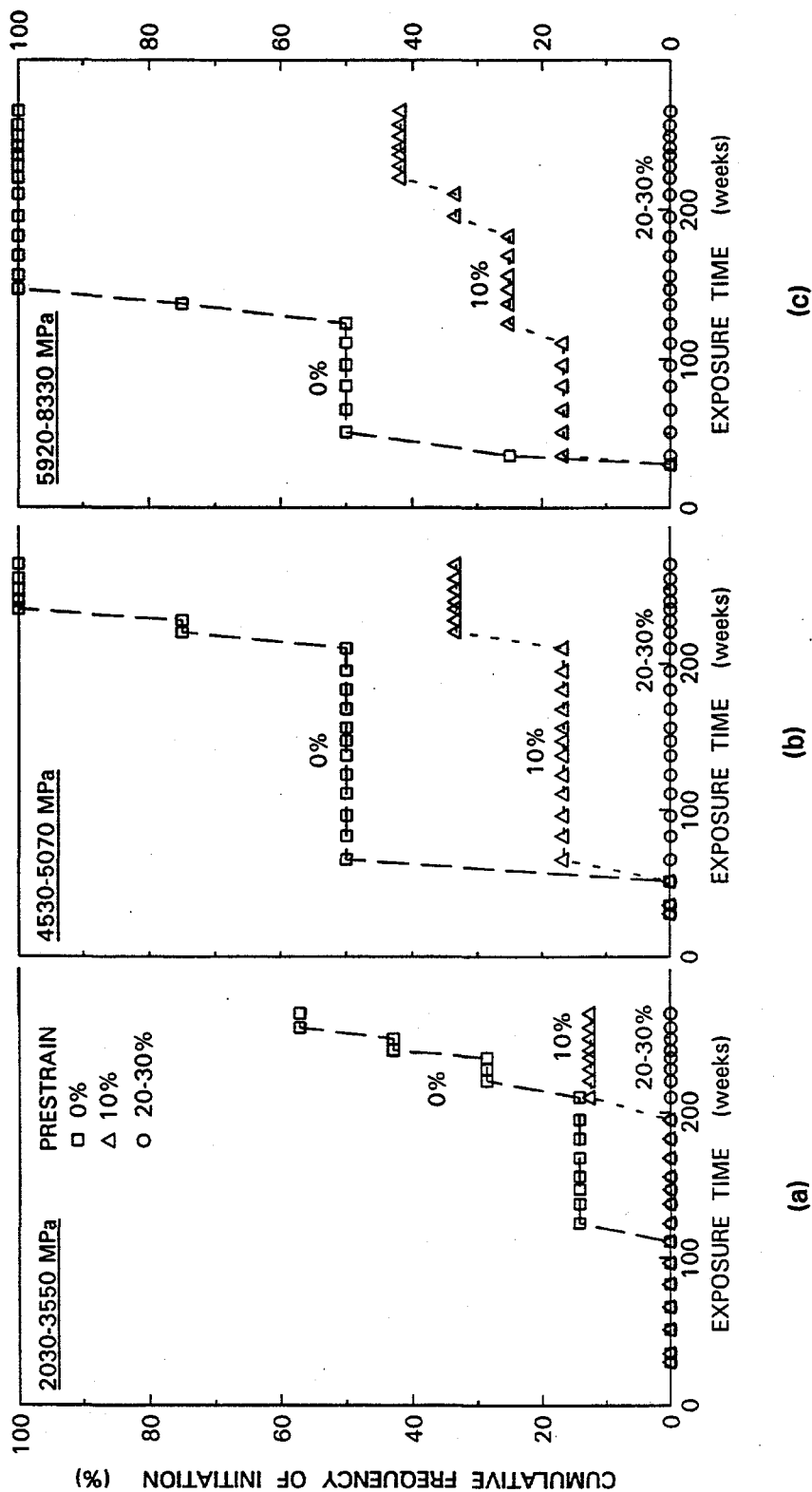


Figure 4. Cumulative frequency of SCC initiation for as-notched specimens from Condition HTH Heat A6 that were prestrained 0%, 10% and 20-30%. (a) Final PSES values of 2030-3550 MPa. (b) Final PSES values of 4530-5070 MPa. (c) Final PSES values of 5920-8330 MPa.

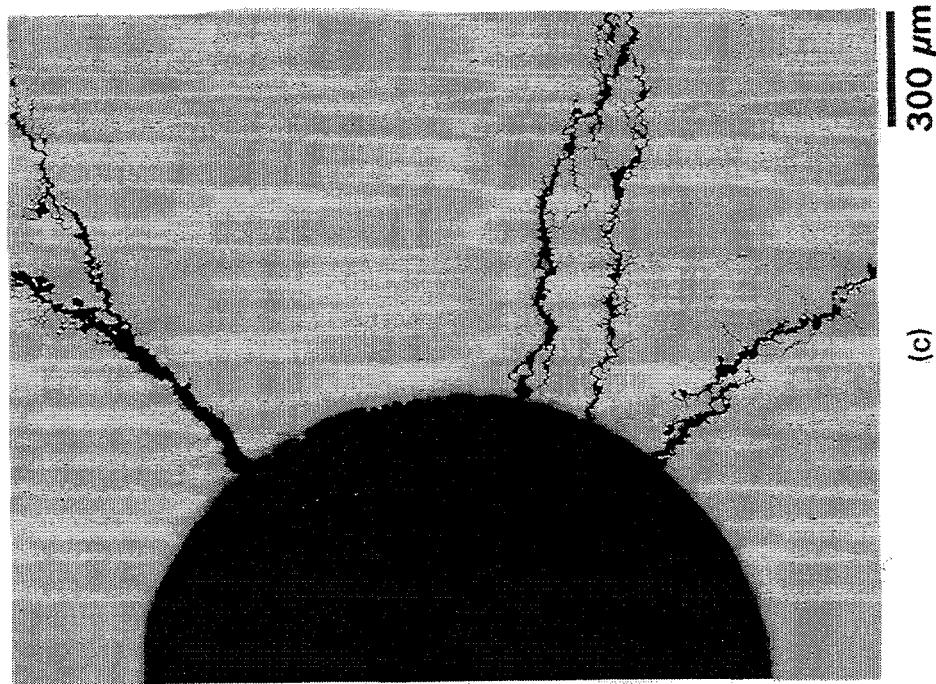
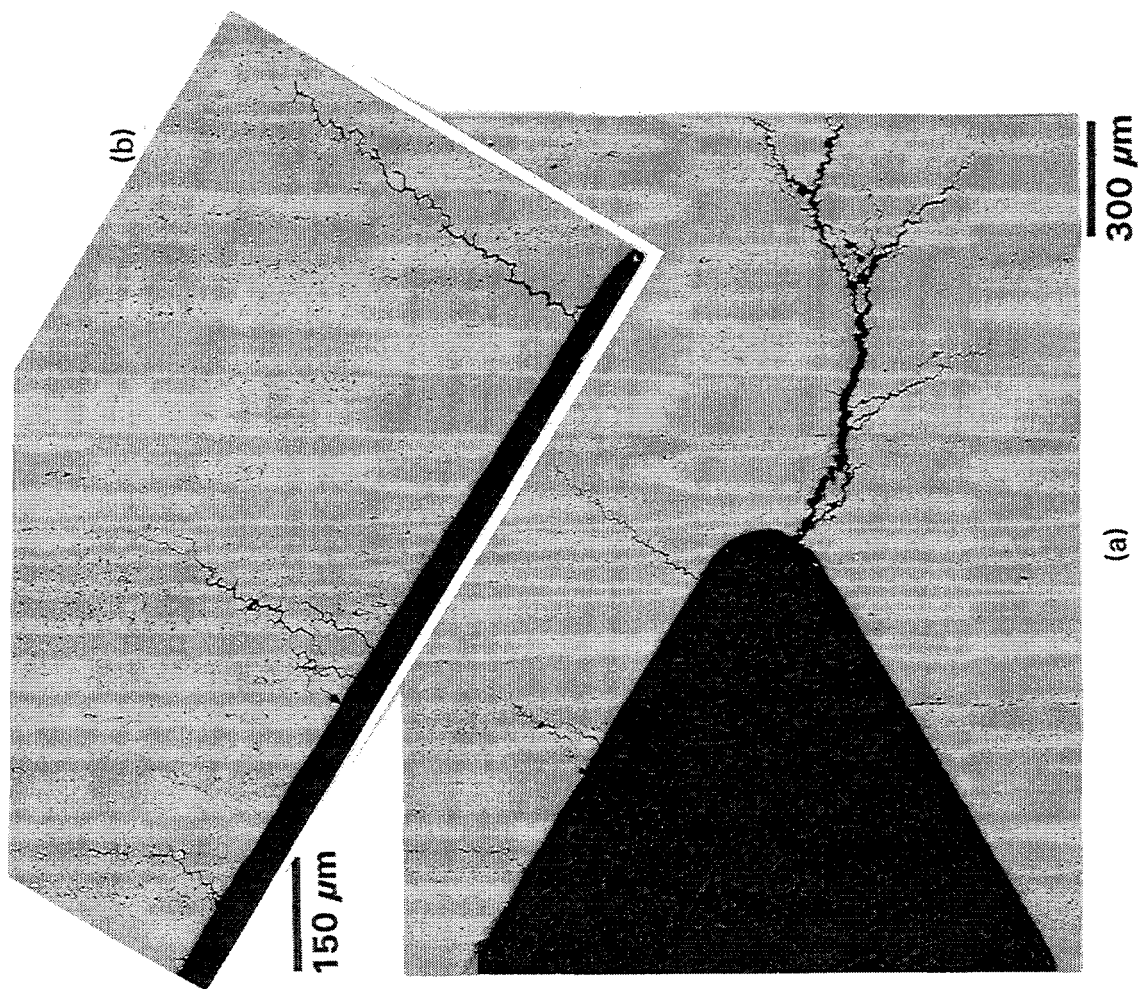


Figure 5. Off-root SCC initiation in as-notched Condition AH specimens. (a) Dominant crack initiated near notch tip ($\rho = 0.1$ mm), but three additional cracks initiated along notch flank. (b) High magnification of cracking along notch flank. (c) Evidence of off-root cracking in blunt notch specimen ($\rho = 0.8$ mm).

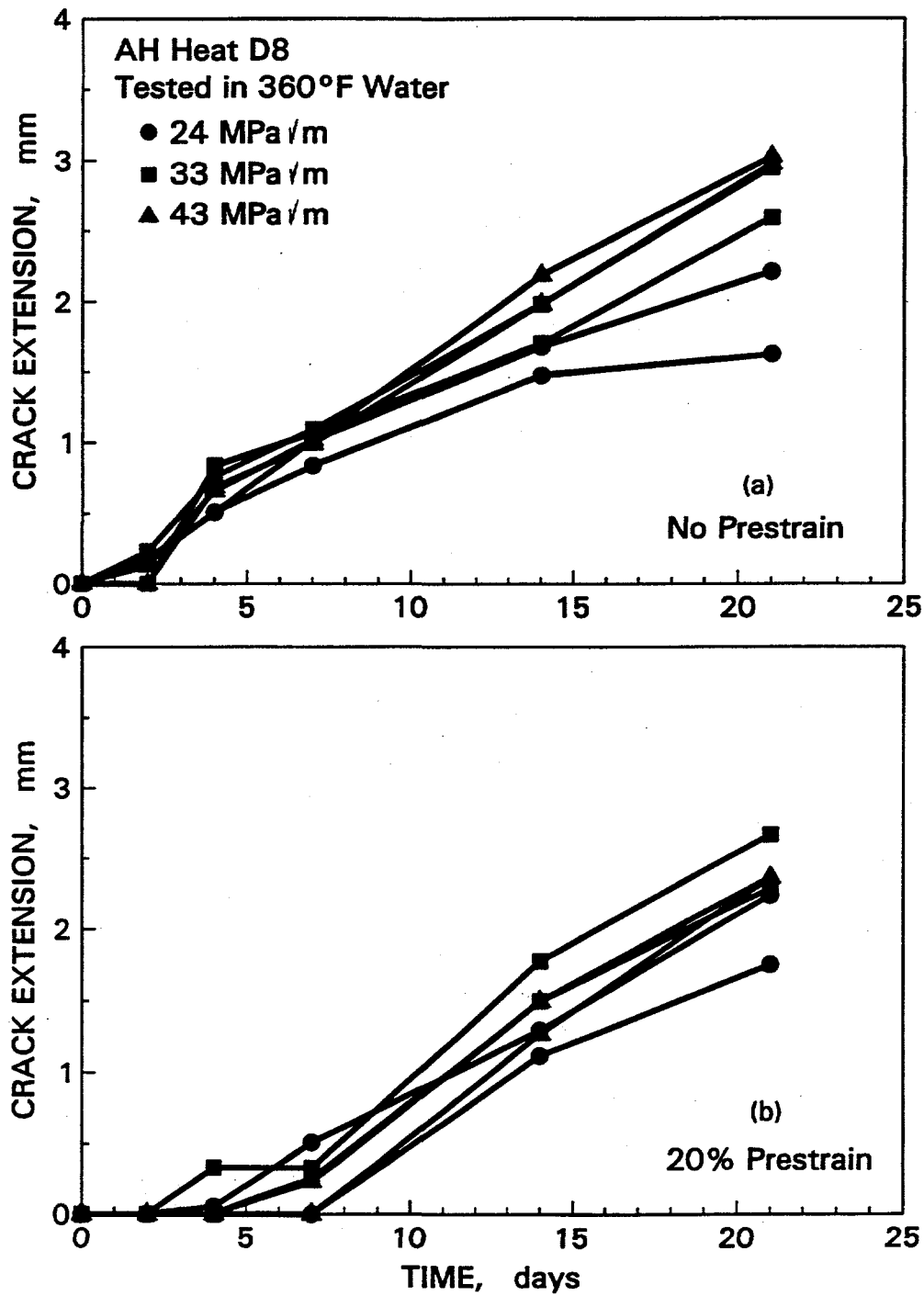


Figure 6. Comparison of SCC behavior in replicate AH specimens at three different K_I levels. (a) No prestrain. (b) 20% prestrain.

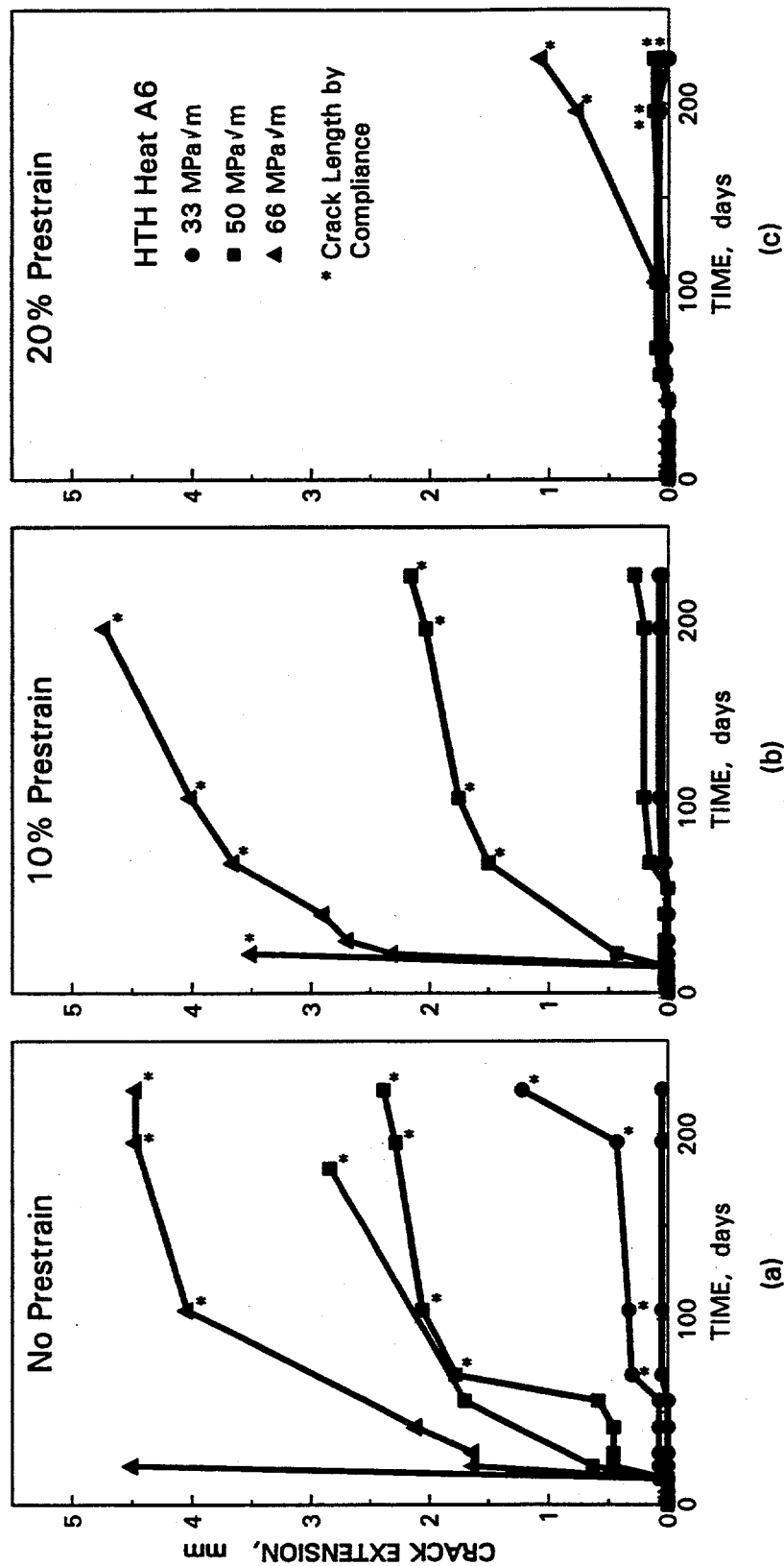


Figure 7. Comparison of SCC behavior for replicate HTH Heat A6 specimens at three different K_I levels. Data points denoted by asterisks indicate that crack extension values were inferred from residual bolt-load measurements. Other values were based on surface crack length measurements. (a) No prestrain. (b) 10% prestrain. (c) 20% prestrain.

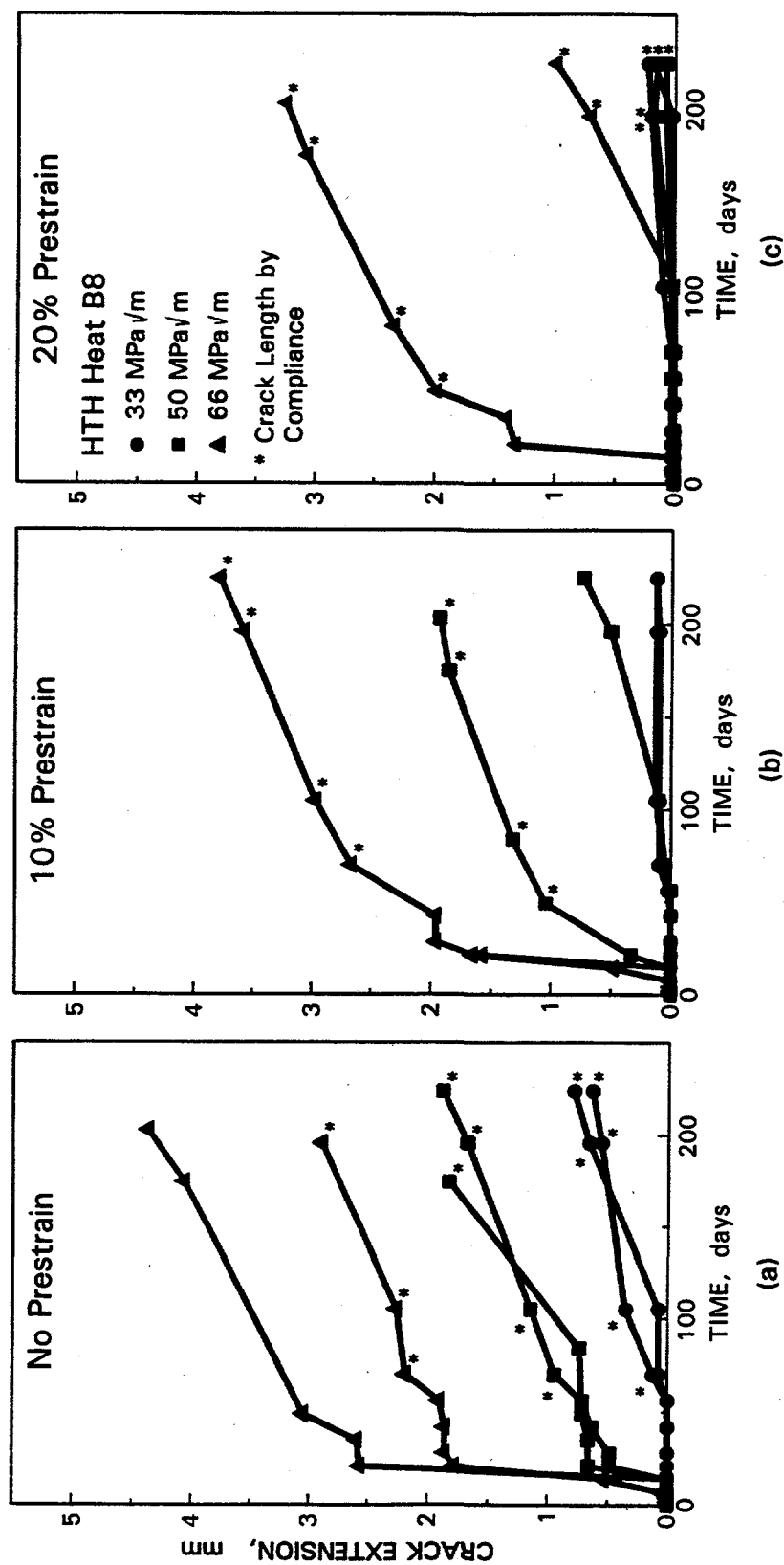


Figure 8. Comparison of SCC behavior for replicate HTH Heat B8 specimens at three different K_I levels. Data points denoted by asterisks indicate that crack extension values were inferred from residual bolt-load measurements. Other values were based on surface crack length measurements. (a) No prestrain. (b) 10% prestrain. (c) 20% prestrain.

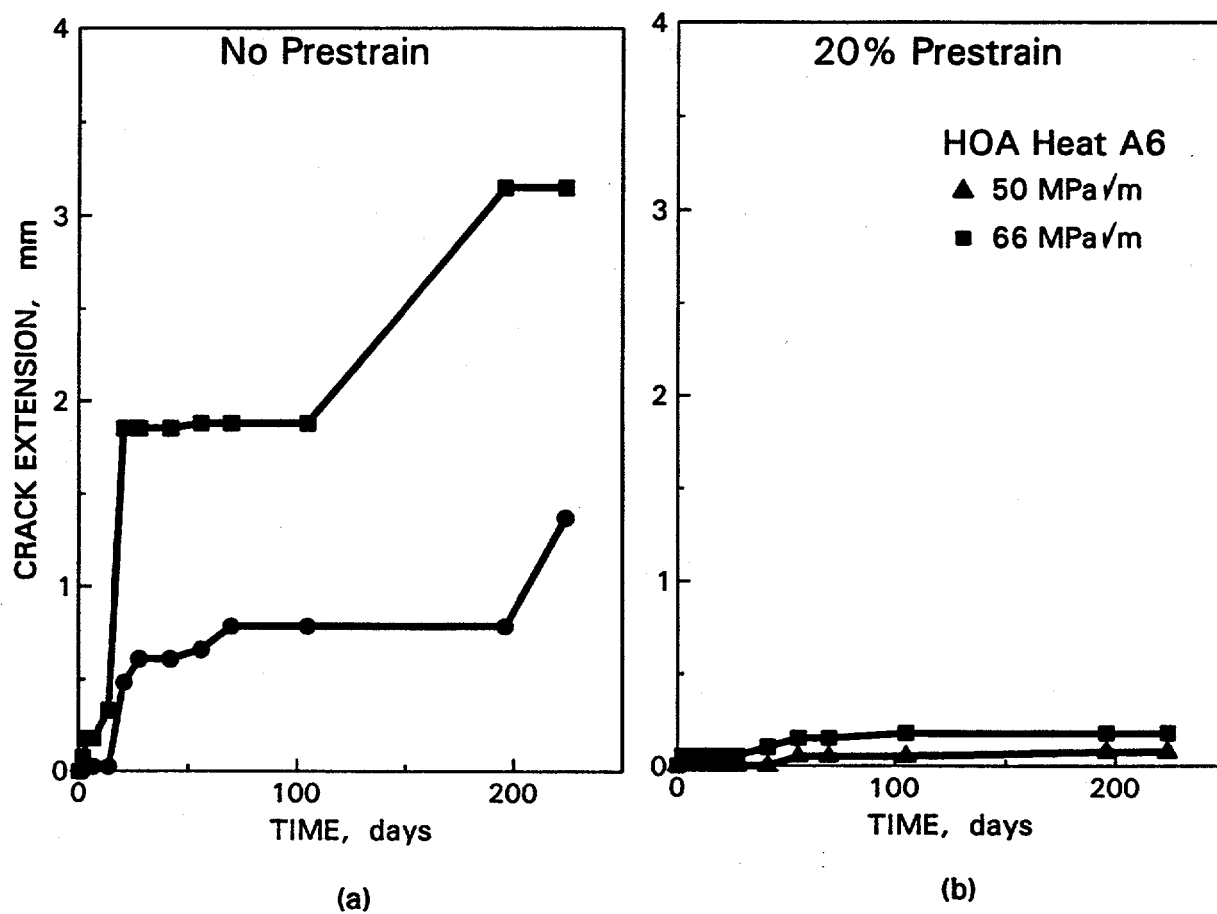


Figure 9. Comparison of SCC behavior for HOA Heat A6 specimens at two different K_I levels. (a) No prestrain. (b) 20% prestrain.

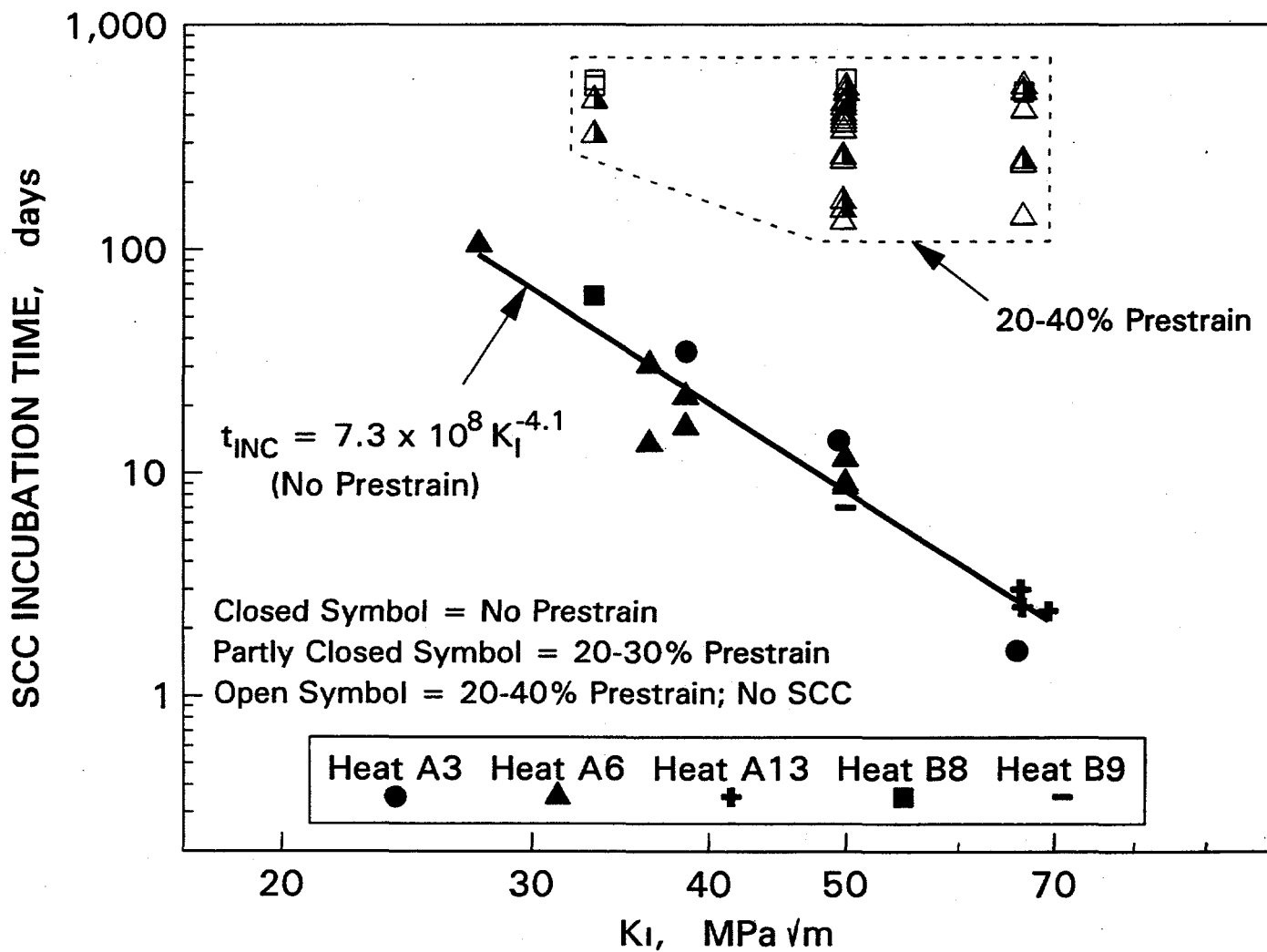


Figure 10. Effect of prestrain on SCC incubation in precracked specimens of HTH Heats A6 and B8. The data for nonprestrained specimens and the associated power-law relationship are from Reference (1). All prestrained specimens were destructively examined; those showing no SCC are represented by open symbols.

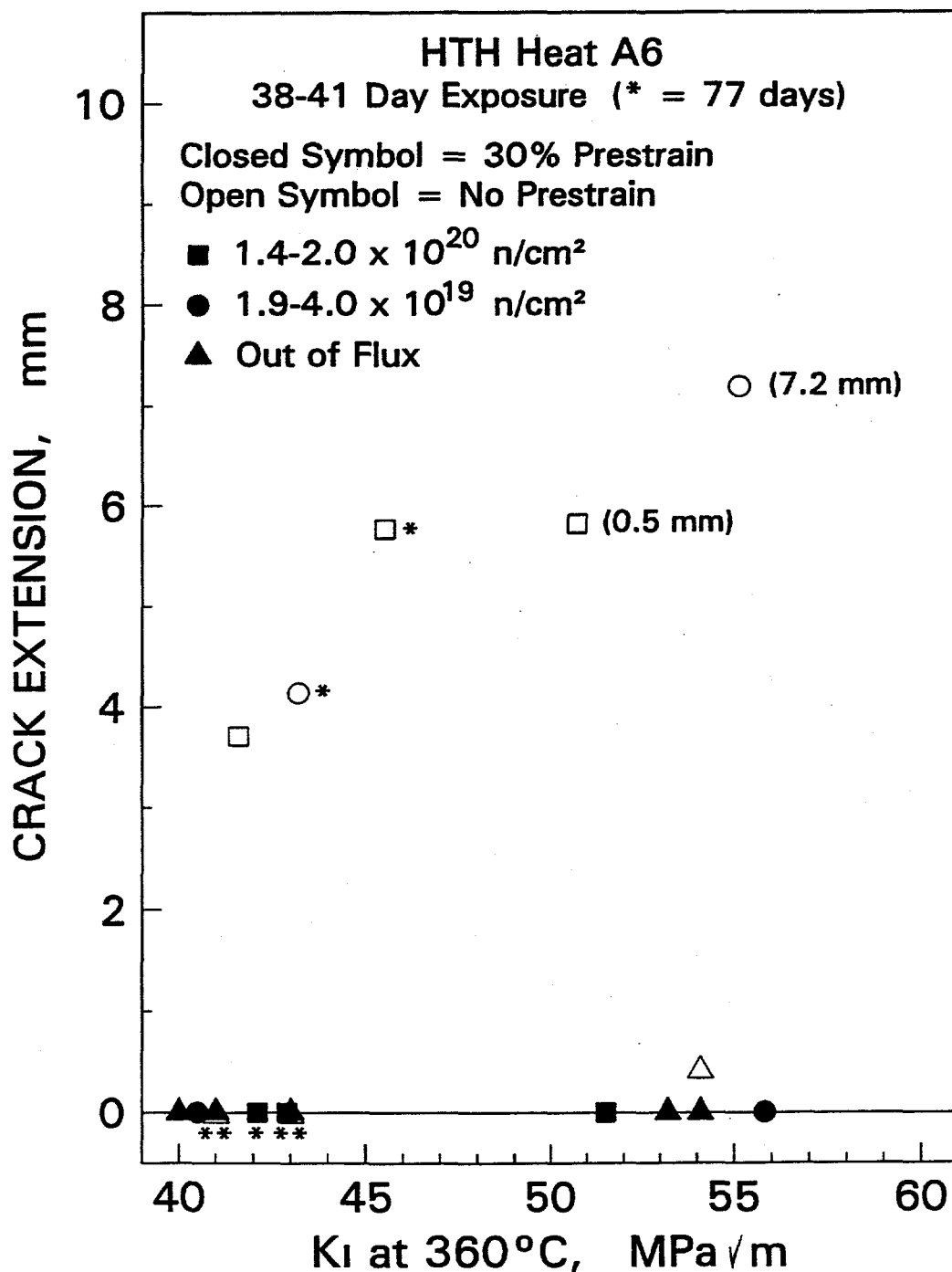


Figure 11. In-reactor performance of nonprestrained and 30% prestrained specimens from HTH Heat A6 in ~360°C water. Most of the specimens were irradiated for 38 to 41 days, while some (denoted by asterisk) were irradiated for 77 days. Crack extension values represent the total amount of cracking that occurred at both high and low temperatures. The amount of low temperature cracking is provided inside parentheses.