# Hydrogen Pickup and Ring-Compression Ductility for E110 Oxidized in Steam at 1000˚C

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## 1. Introduction

ANL has performed steam oxidation tests at 1000˚C, hydrogen analysis, and post-oxidation ductility tests at room temperature with E110 tubing samples. The focus of this work has been on the evolution of oxide layer instability and hydrogen pickup during two-sided, steam oxidation at 1000˚C. These results are documented in Ref. 1. A limited number of room-temperature ring-compression tests have been conducted with these samples in an effort to correlate loss in ductility with hydrogen pickup. The 8-mm-long ring-compression samples are cut from the 25-mm-long oxidation samples. Ring-compression tests are conducted at a cross-head displacement rate of 2 mm/minute. As these samples experienced significant breakaway oxidation, partial-to-full delamination of the oxide layer and partial spallation of the oxide layer, the measured change in sample weight represents a lower bound on the degree of oxidation. At 1000˚C, black oxide forms on E110 at a rate slower than predicted by the Cathcart-Pawel model. However, white-to-gray oxide forms at a rate faster than predicted by the same model. Ductility results are compared to the Cathcart-Pawel model predictions for ECR, which are likely a better indication of ECR in the range of 0-7% than the values based on change in sample weight.

## 2. Results

Table 1 summarizes the results for hydrogen pickup and post-oxidation ductility (see Appendix A) for as-received E110 samples oxidized at 1000˚C and furnace cooled. For hold times at 1000˚C between ≈300 and ≈600 s, there is a transition between ductile and brittle behavior. Samples with ≤2% offset strain are assumed to be brittle. The oxide layer has already experienced local breakaway oxidation by ≈300 s resulting in an average hydrogen pickup of 120 wppm. By ≈600-s hold time, the extent of the sample surfaces experiencing breakaway oxidation has increased, resulting in local variation in hydrogen pickup from 25 to 560 wppm. The average value for the sample tested at ≈600-s hold time is 275 wppm. E110 appears to lose its ductility with hydrogen pickup in the range of 120 to 275 wppm (see Fig. 1). The corresponding calculated ECR for embrittlement is ≈7% at 1000˚C (see Fig. 2).

The critical hydrogen level for embrittlement of E110 at 1000˚C cannot be determined precisely from the data in Table 1 because of the wide variation in hydrogen content within a single sample. However, if Test EU#38 is repeated and the ring-compression sample were to be prepared from the center of the oxidation sample, it is estimated that the average hydrogen content would be 125 wppm with a local variation of 25-285 wppm. Such a sample may exhibit some ductility. If it does, then a second test should be conducted on a sample with a hold time of 714 s and a calculated ECR of 7.6%. These two additional tests would help to better define the critical hydrogen level for E110 embrittlement following steam oxidation at 1000˚C.

Table 1 Ring Compression Test Results for E110 Samples Oxidized at 1000˚C and Slow Cooled. Tests were performed on 8-mm-long samples at room temperature and at 2 mm/minute cross-head displacement rate.

| Test ID | Hold Time s | ECR, % CP-Model | ECR, % Measured | H Content (Variation) wppm | Ring Sample Location (0 mm = middle) | RT Off-Set Displacement mm | RT Ductility % |
|---|---|---|---|---|---|---|---|
| E110 | 0 | 0 | 0 | 3 | --- | 6 | 66 |
| EU#10 | 5 | 1.8 | ≥1.3 | --- | --- | 6.05 | 66 |
| EU#9 | 290 | 5.0 | >3.7 | --- | --- | --- | --- |
| EU#12 | 290 | 5.0 | >3.3 | 120 (70-170) | --- | 5.44 | 59 |
| EU#36 | 395 | 5.8 | >3.8 | 32 (4-84) | -10 mm to -2 mm | 5.82 | 63 |
| EU#38 | 625 | 7.1 | >5.6 | 275 (25-560) | -8 mm to 0 mm | 0.13 | 1.4 (brittle) |
| EU#38A | 625 | 7.1 | TBD | ≈125 ≈(25-285) | -4 mm to + 4 mm | TBD | TBD |
| EU#50 | 714 | 7.6 | TBD | TBD | -4 mm to + 4 mm | TBD | TBD |
| EU#40 | 825 | 8.1 | >5.8 | 925 (440-1500) | -8 mm to 0 mm | 0.04 | 0.4 (brittle) |
| EU#8 | 1400 | 10.4 | --- | 4230±250 | --- | 0 | 0 |

**References**


1. Y. Yan, T. Burtseva and M. C. Billone, "Progress Report on E110 Post-Quench Ductility Test Program," letter report to NRC and RRC KI, May 31, 2003.

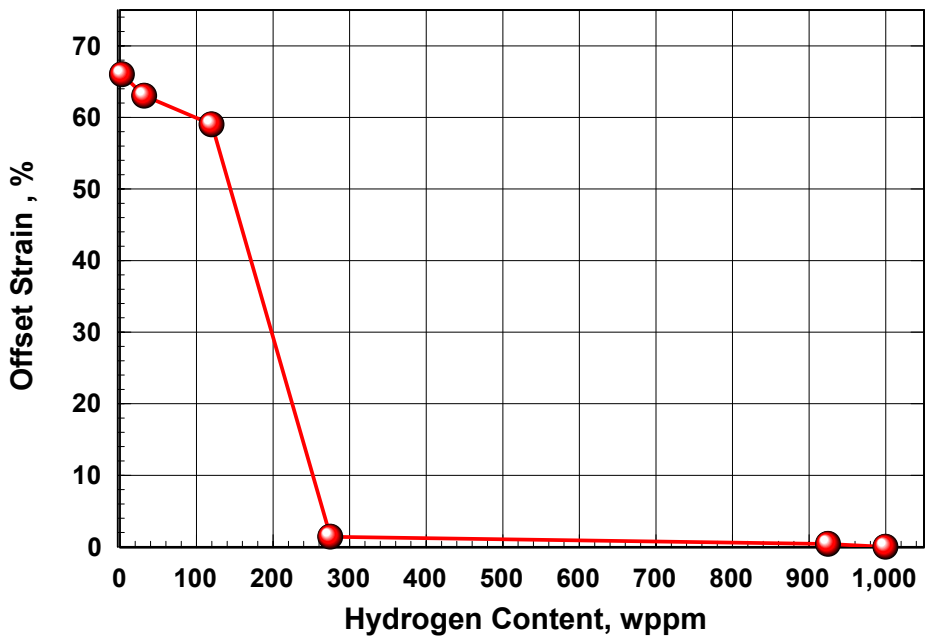


Fig. 1. Room-temperature, ring-compression ductility vs. hydrogen content of as-received E110 tubing oxidized (two-sided) in steam at 1000˚C for hold times of 0-825 s. The sample length is 8 mm and the cross-head displacement is 2 mm/minute.

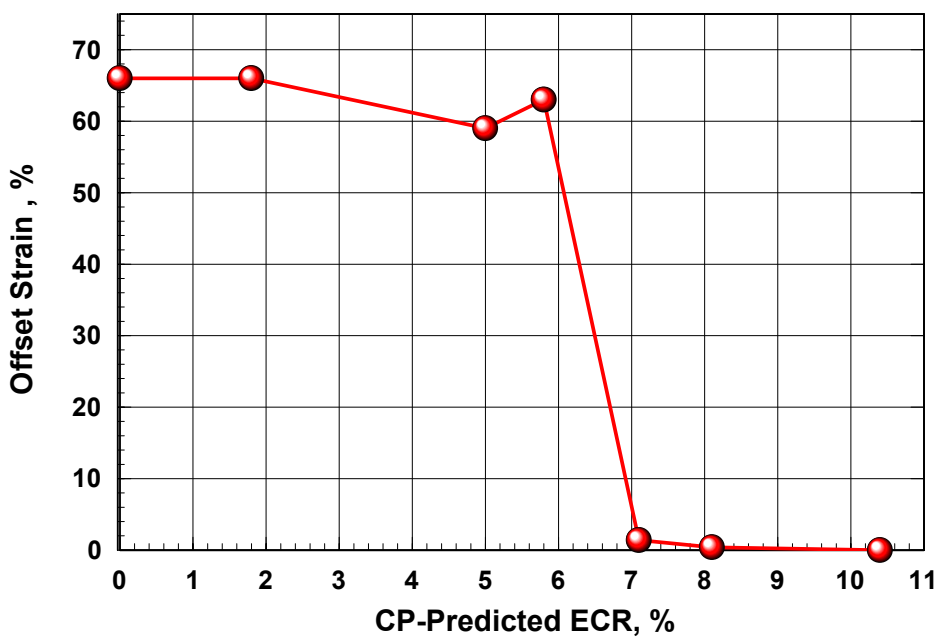


Fig. 2. Room-temperature, ring-compression ductility vs. Cathcart-Pawel (CP) predicted ECR of as-received E110 tubing oxidized (two-sided) in steam at 1000˚C for hold times of 0-825 s. The sample length is 8 mm and the cross-head displacement is 2 mm/minute.

# APPENDIX A

## Load-Displacement Curves for Oxidized and Slow-Cooled E110 Samples Subjected to Ring-Compression Tests at Room Temperature and a Cross-head Displacement Rate of 2 mm/minute

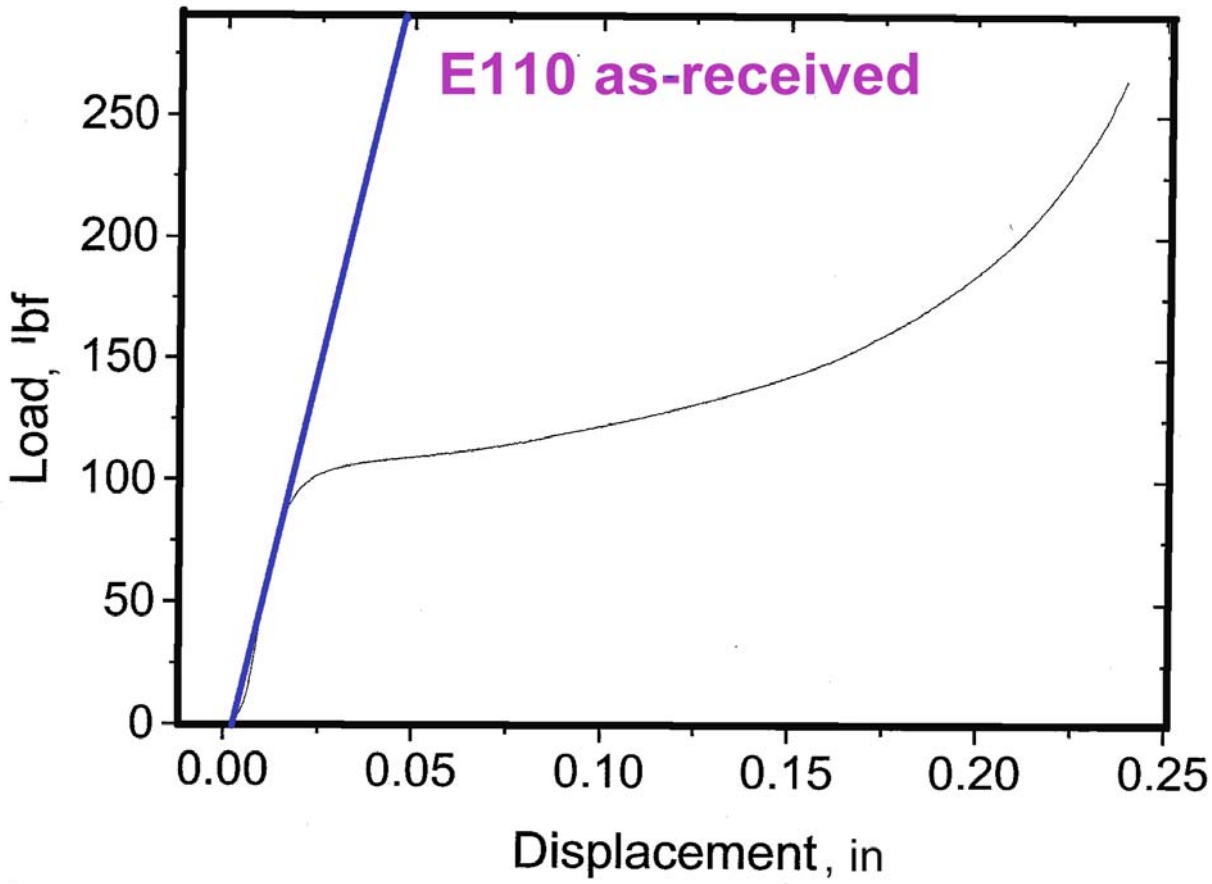


Fig. A.1 Room-temperature ring-compression results for as-received E110 tubing sample (8-mm long, 0.71-mm thick and 9.17-mm outer diameter. Cross-head displacement rate was 2 mm/minute.

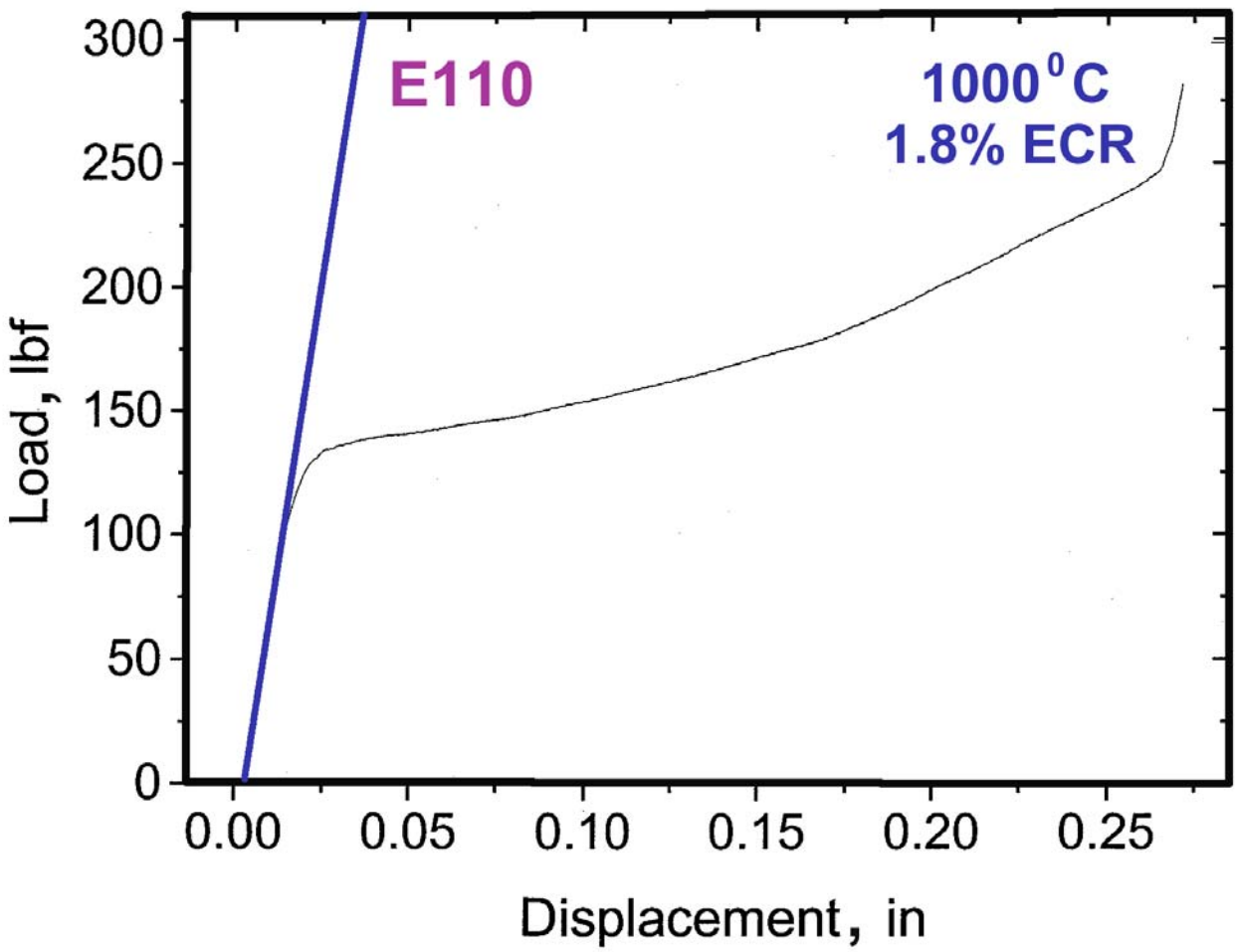


Fig. A.2 Room-temperature ring-compression results for 8-mm-long E110 tubing sample after oxidation at 1000˚C to a calculated ECR of 1.8%. Cross-head displacement rate was 2 mm/minute.

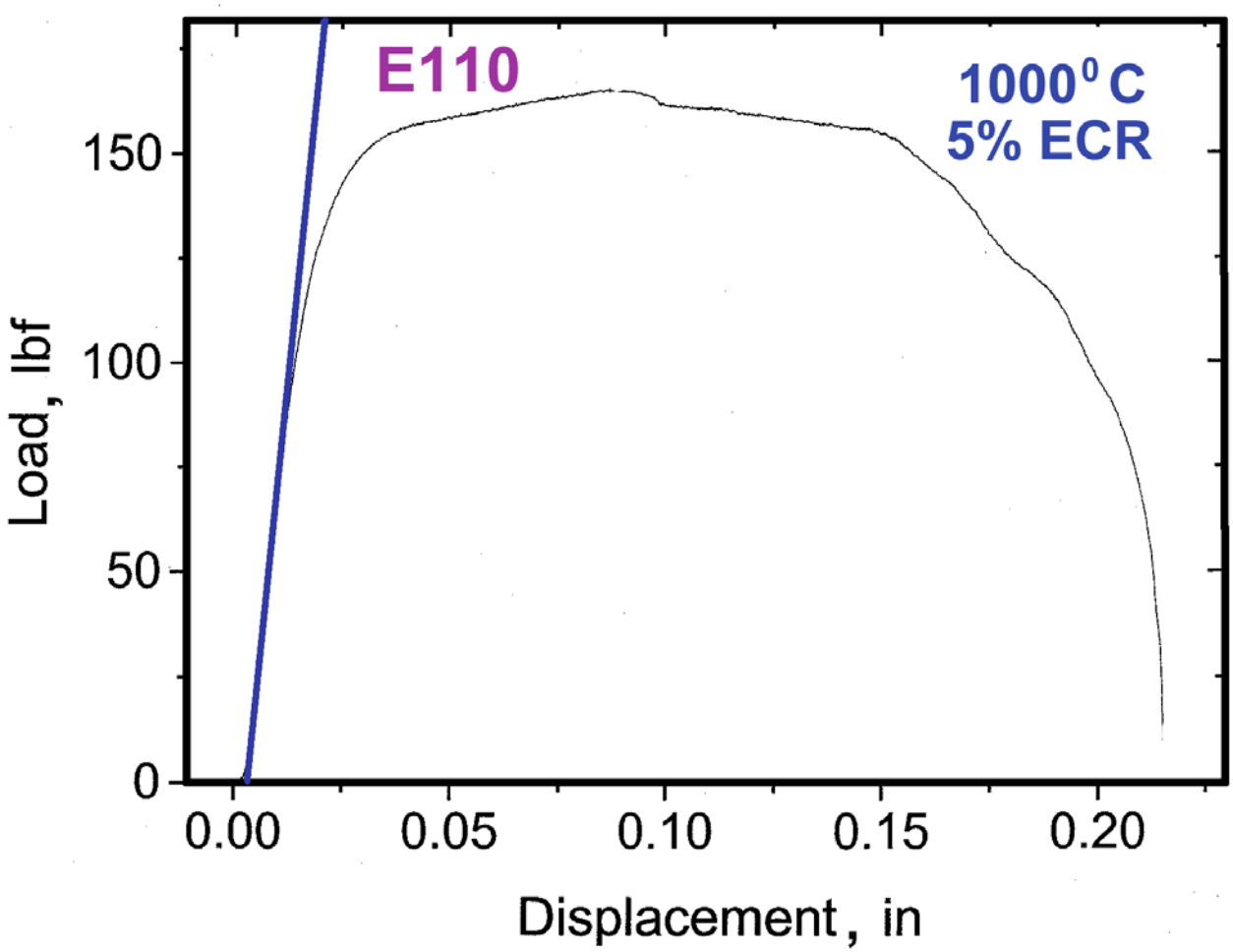


Fig. A.3 Room-temperature ring-compression results for 8-mm-long E110 tubing sample after oxidation at 1000˚C to a calculated ECR of 5%. Cross-head displacement rate was 2 mm/minute.

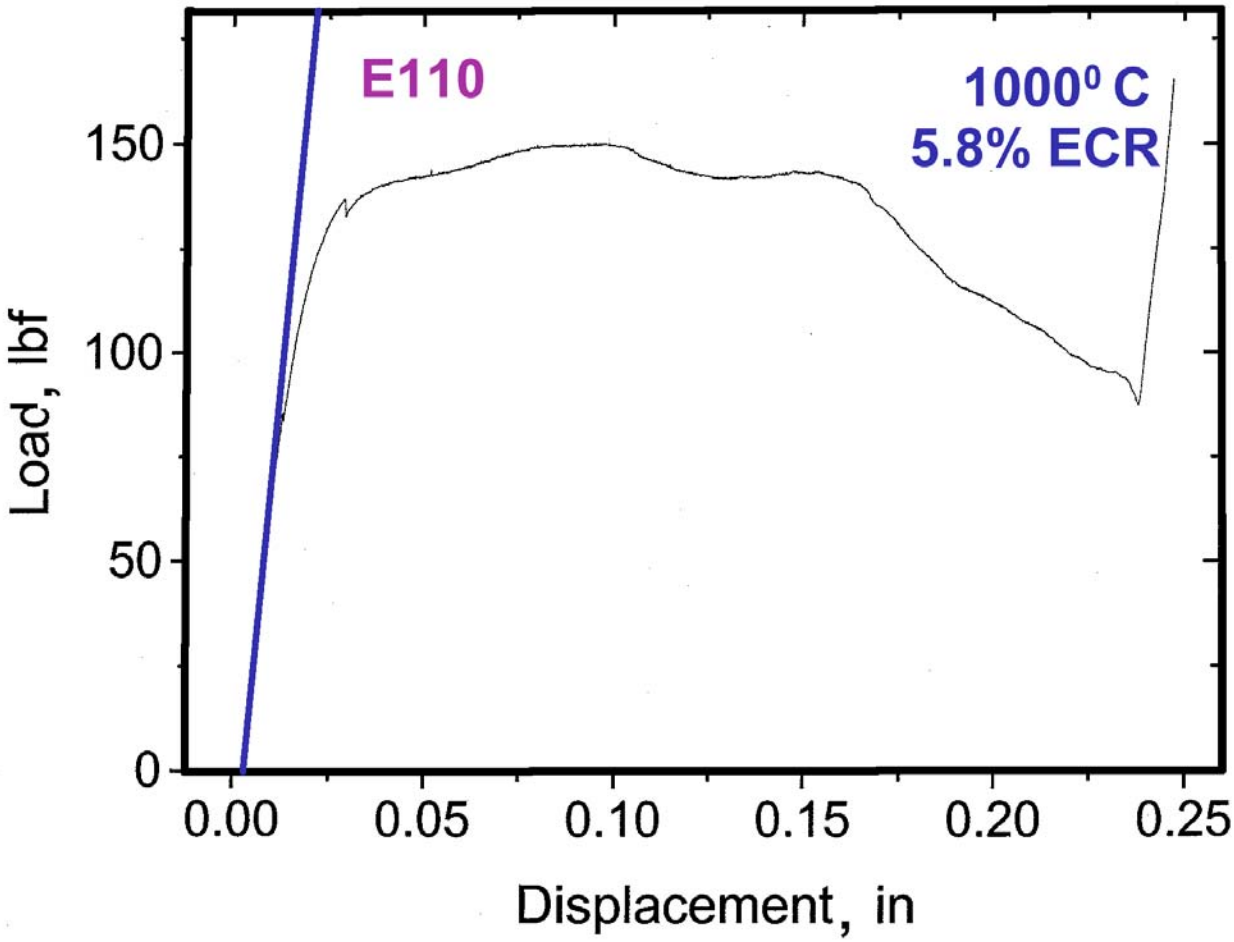


Fig. A.4 Room-temperature ring-compression results for 8-mm-long E110 tubing sample after oxidation at 1000˚C to a calculated ECR of 5.8%. Cross-head displacement rate was 2 mm/minute.

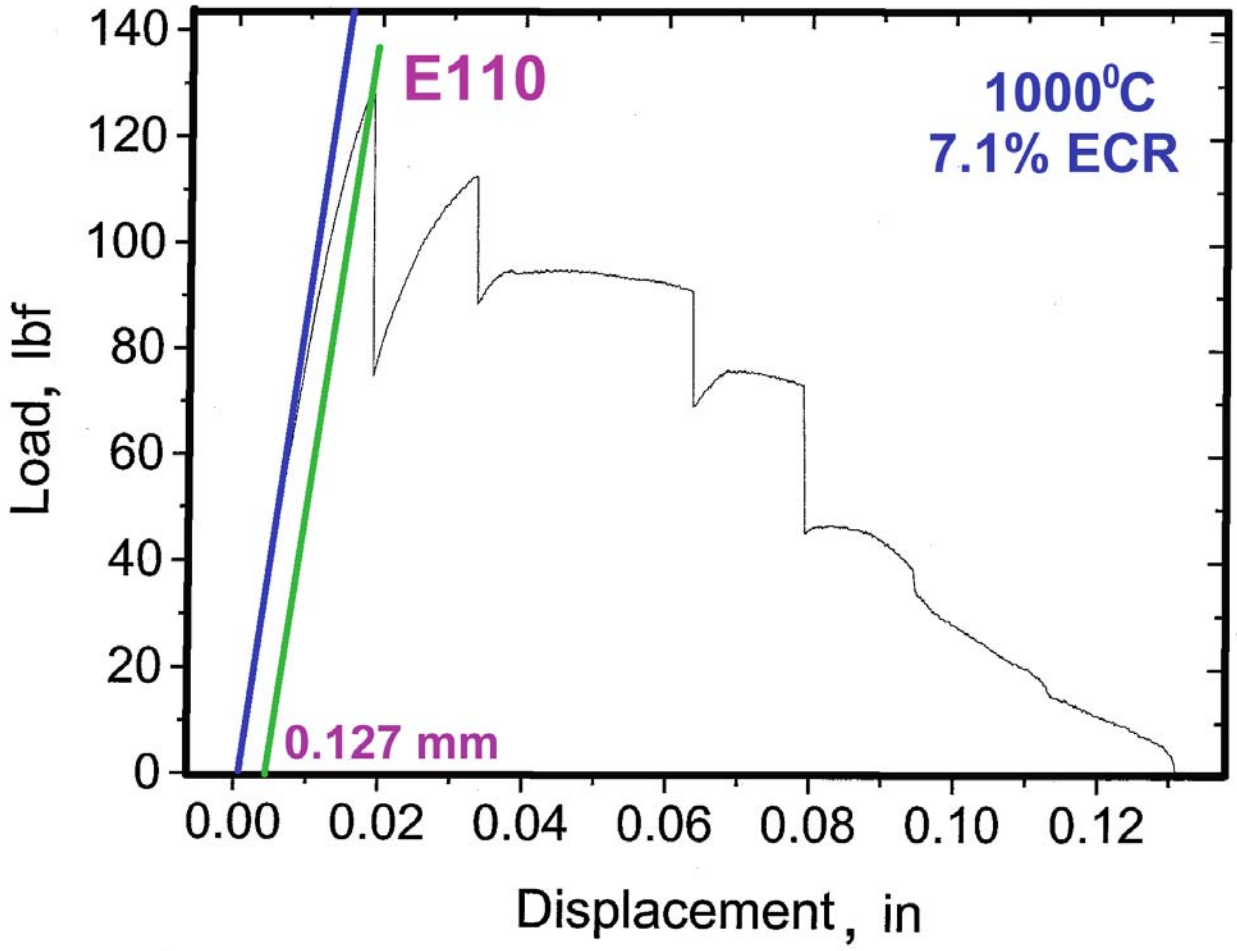


Fig. A.5 Room-temperature ring-compression results for 8-mm-long E110 tubing sample after oxidation at 1000˚C to a calculated ECR of 7.1%. Cross-head displacement rate was 2 mm/minute.

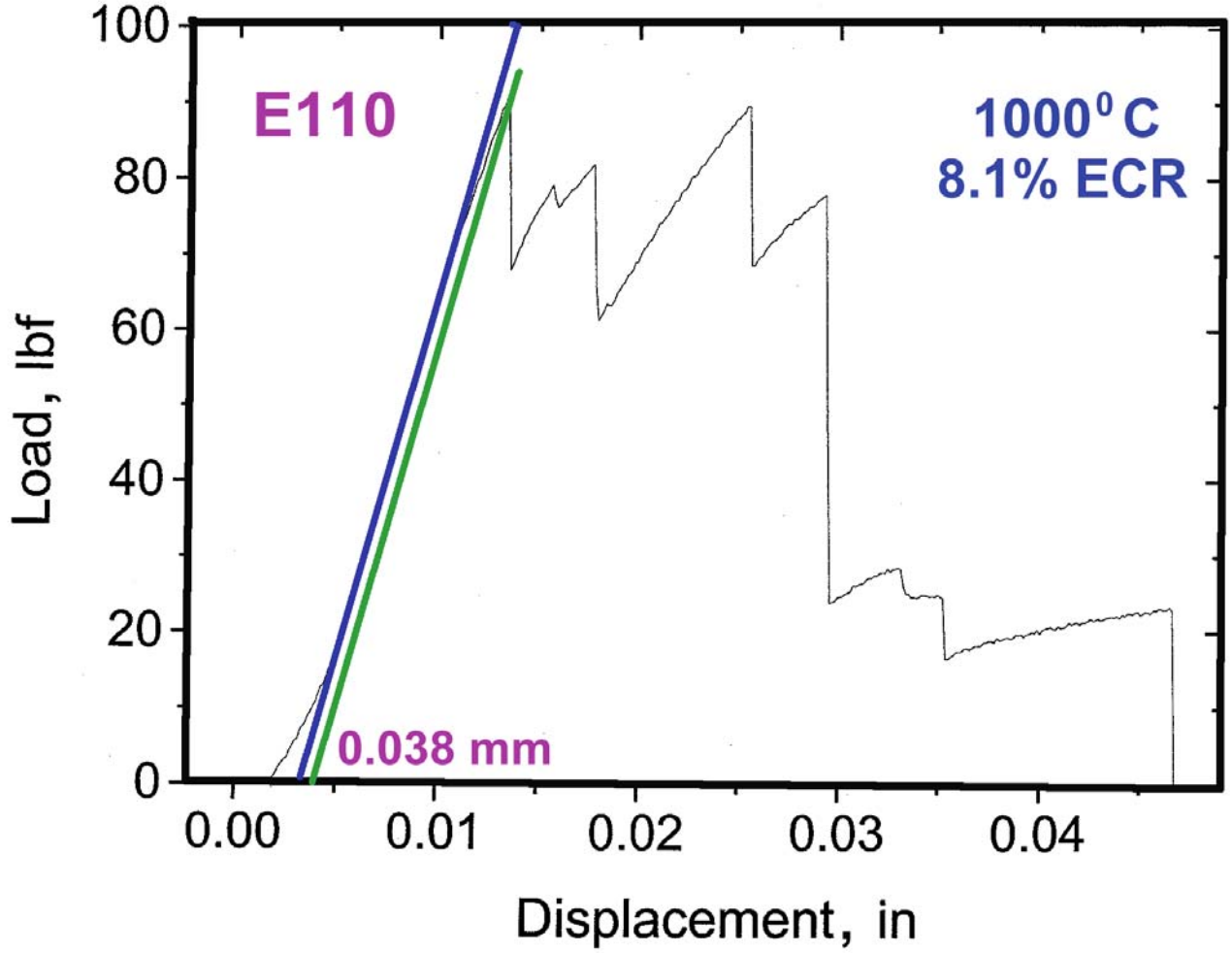


Fig. A.6 Room-temperature ring-compression results for 8-mm-long E110 tubing sample after oxidation at 1000˚C to a calculated ECR of 8.1%. Cross-head displacement rate was 2 mm/minut