The Strain Rate Dependent Plastic Flow Behavior of Zirconium and its Alloys

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The effect of strain rate \((10^{-5} \text{ to } 10^{-1} \text{ min}^{-1})\) on the plastic flow behavior was examined by means of tension tests over a range of temperatures \((RT \sim 500^\circ C)\). The principal material examined was Zircaloy-2, and the others were iodide zirconium and binary alloys of Zr-0.1 pct O and Zr-1.5 pct Sn by weight. In Zircaloy-2, the behavior was characterized by a sudden increase of flow strength with decreasing strain rate; concurrently the ductility decreased. Other interrupted and hold time experiments showed that the process is essentially that of a strain rate-induced strengthening phenomenon, with several features which could be identified with the strain aging process. It was further shown that the critical range of strain rates and temperatures at which the anomalous behavior took place could be correlated with the minimum in the strain rate sensitivity of flow stress. A similar but less pronounced flow behavior was observed with pure zirconium and its binary alloys, but with no marked change in the tensile ductility. From these results, the role of oxygen and tin on the early stage of plastic flow behavior was discussed in terms of dislocation-impurity interaction mechanisms. It was however concluded that these elements are not directly responsible for the ductility loss at slow strain rate in Zircaloy-2.

In recent years it has become increasingly evident that zirconium and some of its alloys exhibit an anomalous mechanical behavior over the temperature range of approximately 200°C to 400°C. The observed phenomena in this temperature range include the yield-point effect and the flow stress increase after interrupted loading in tension testing,\(^{15,16}\) the decreasing creep rate with increasing temperature,\(^{11-13}\) and the decreasing tensile ductility with both increasing temperature\(^ {15,14}\) and decreasing strain rate.\(^ {15}\) Many of these phenomena have been related to some form of strain aging, and a variety of mechanisms have been proposed. For example, the effects have been associated with the precipitation of tin in Zr-1.5 Sn alloy,\(^ {2}\) with hydrogen segregation in Zircaloy-2,\(^ {3}\) with the Cottrell locking of dislocations by impurity atmosphere,\(^ {13}\) or with the other dislocation-interstitial interactions, such as with oxygen.\(^ {5,6}\) The dynamic dislocation model for yielding also has been explained to yield the yield-point behavior.\(^ {6}\)

Other related anomalies in these alloys have also been reported. It was found that the apparent activation energy in creep increased abruptly followed by a sharp drop at about 350°C.\(^ {13,16}\) This was again attributed to a solute-dislocation interaction. Another notable characteristic is that of the sharply lower strain rate sensitivity of the flow stress in the temperature range of 300°C to 400°C in pure zirconium,\(^ {7}\) Zircaloy-2,\(^ {3}\) and Zircaloy-4.\(^ {17}\) Similar minima in strain rate sensitivity were also observed in single crystals both of Zircaloy-2\(^ {18,19}\) and zirconium oxygen alloys.\(^ {10,21}\)

For some of the widely used complex commercial alloys like Zircaloy-2, this anomalous behavior has not yet been adequately characterized. The multiplicity of alloying elements in addition to tin and oxygen in such alloys is the reason for this lack of understanding. It is therefore important that the nature of this anomalous behavior be identified and also that the flow-stress behavior up to and including fracture be characterized.

An attempt was made in this work to establish the phenomenology of the stress-strain rate-temperature relationship in Zircaloy-2 as well as the strain-rate dependence of the fracture strain. Also examined were the effects of alloying elements such as oxygen and tin in zirconium in amounts comparable to those found in Zircaloy-2. The mechanical behavior of these alloys were in turn compared with that of pure zirconium, where the structure and testing conditions were comparable.

**Experimental Procedure**

**Processing and Structures**

Both iodide grade zirconium and Zircaloy-2 were obtained in the form of forged ingots. The binary alloys of oxygen and tin were prepared by remelting the same iodide grade zirconium using a vacuum arc melting method. The details of the subsequent thermal-mechanical processing schedule are given in Table I; the final recrystallization treatment was designed to produce comparable grain sizes in all the materials. The microstructures of the recrystallized materials are shown in Fig. 1. The results of the chemical analyses made after the final heat treatment are shown in Table II.

The crystallographic texture was determined by the Siemens' pole-figure device, described elsewhere.\(^ {18}\) The pole figure of Zircaloy-2 was nearly the same as that of Schedule J in the previous work,\(^ {15}\) i.e., the (0002) pole was tilted about 35 deg from the sheet normal toward the transverse direction. A similar texture was found for the iodide grade zirconium. The texture of the binary alloy sheets was not determined since the measurements of the transverse strain in
tension indicated a behavior similar to the pure zirconium.

Tensile Specimens and Testing

The orientation of all specimens was such that the specimen axis was parallel to the transverse direction in the sheet. The gage section was 0.2 by 0.055 in. and 0.8 in. long. A strain extensometer was used over a 0.5 in. portion of the gage length in all the tests.

All tests were conducted in an Instron machine. At elevated temperatures, tests were made in an Instron chamber containing a helium atmosphere. Three types of tests were used: monotonic straining to fracture at a fixed crosshead speed, interrupted straining where prestrain, hold time, and crosshead speed were varied, and finally, differential strain rate tests. In the latter tests, the crosshead speed was varied from $2 \times 10^{-3}$ in. per min to 2 in. per min in a manner shown schematically in Fig. 2. Since the material always strain hardens, the load extrapolation method shown in Fig. 2 was used in the differential strain rate tests to obtain a

Table I. Processing History and Material Details

<table>
<thead>
<tr>
<th>Material</th>
<th>Initial Condition</th>
<th>Primary and Final Working</th>
<th>Final Treatment</th>
<th>Grain Size, $\mu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iodide grade Zr</td>
<td>3/4 in. thick plate cut from a forged ingot</td>
<td>Hot roll to 0.140 in. in thickness at 400°C and rolled at RT to 0.062 in.</td>
<td>600°C/25 min</td>
<td>9.5</td>
</tr>
<tr>
<td>Zr + 0.1 pct O</td>
<td>Vacuum arc melted and machined disc of 0.320 in. in thickness</td>
<td>Upset at 930°C to 0.220 in. in thickness and rolled from 400°C to 0.062 in.</td>
<td>640°C/1½ hr</td>
<td>9.4</td>
</tr>
<tr>
<td>Zr + 1.5 pct Sn</td>
<td>Vacuum arc melted and machined disc of 0.320 in. in thickness</td>
<td>Upset at 930°C to 0.220 in. in thickness and rolled from 400°C to 0.062 in.</td>
<td>640°C/1½ hr</td>
<td>8.5</td>
</tr>
<tr>
<td>Zircaloy-2</td>
<td>1 in. thick plate cut from a forged ingot</td>
<td>Hot roll from 900°C to 0.160 in. in thickness followed by cold rolling to 0.062 in. in thickness</td>
<td>750°C/45 min</td>
<td>11.0</td>
</tr>
</tbody>
</table>

Table 1—Representative microstructures as in the recrystallized condition: (a) Iodide grade zirconium, (b) Zr-O alloy, (c) Zr-Sn alloy, and (d) Zircaloy-2. Prior to the final mechanical polish the specimens were etched in a solution of 2.5 parts HF, 22 parts HNO$_3$, and 45 parts lactic acid with 22 parts water. Rolling direction is vertical.