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PREFACE

More than 2,100 papers were submitted by the nations, the specialized agencies and the International Atomic Energy Agency, which participated in the Second United Nations International Conference on the Peaceful Uses of Atomic Energy. The number of papers was thus about twice that involved in the First Conference. Provision was therefore made to hold five concurrent technical sessions in comparison with the three that were held in 1955. Even so, the percentage of orally presented papers was less in 1958 than in 1955.

In arranging the programme, the Conference Secretariat aimed at achieving a balance, allowing adequate time for presentation of as many papers as possible and, nevertheless, leaving time for discussion of the data presented. Three afternoons were left free of programme activities so that informal meetings and discussions among smaller groups could be arranged. No records of these informal meetings were made.

A scientific editorial team assembled by the United Nations checked and edited all of the material included in these volumes. This team consisted of: Mr. John H. Martens, Miss L. Ourom, Dr. Walter M. Barss, Dr. Lewis G. Bassett, Mr. K. R. E. Smith, Martha Gerrard, Mr. F. Hudswell, Betty Guttman, Dr. John H. Pomeroy, Mr. W. B. Woollen, Dr. K. S. Singwi, Mr. T. E. F. Carr, Dr. A. C. Kolb, Dr. A. H. S. Matterson, Mr S. Peter Welgos, Dr. I. D. Rojanski, Dr. David Finkelstein, Dr. Cavid Erginsoy (Dr. Erginsoy's services were furnished through the courtesy of the International Atomic Energy Agency), Dr. Vera J Peterson, Dr. Paul S. Henshaw, Dr. Hywell G. Jones, Dr. Alvin Glassner and Mr. J. W. Greenwood.

The speedy publication of such a vast bulk of literature obviously presents considerable problems. The efforts of the editors have therefore been primarily directed towards scientific accuracy. Editing for style has of necessity been kept to a minimum, and this should be noted particularly in connection with the English translations of certain papers from French, Russian and Spanish.

The Governments of the Union of Soviet Socialist Republics and of Czechoslovakia provided English translations of the papers submitted by them. Similarly, the Government of Canada provided French-language versions of the Canadian papers selected for the French edition. Such assistance from Governments has helped greatly to speed publication.

The task of printing this very large collection of scientific information has been shared by printers in Canada, France, Switzerland, the United Kingdom and the United States of America.

The complete Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy are published in a 33-volume English-language edition as follows:

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Recent Advances in the Metallurgy of Zirconium and Titanium Alloys of Special Interest in Reactor Technology

By G. M. Adamson Jr., J. O. Betterton Jr., J. H. Frye Jr. and M. L. Picklesimer *

At the time of the first Geneva Conference, the status of zirconium and titanium metallurgy was such that the metals and certain dilute alloys could be employed effectively in simple structures, which did not require exceptional mechanical properties, and in environments that were not extremely corrosive. The principal advances since that time have been as follows:

1. The fabrication of Zircaloy-2 has been advanced by the development of a fabrication schedule which produces more nearly isotropic plate.

2. Welding procedures have been developed which permit the field construction of complex structures of alpha titanium, and perhaps zirconium, alloys.

3. Zirconium alloys have been developed which are heat treatable to high strengths with moderate ductilities.

4. The hazards associated with the use of zirconium and titanium alloys in reactors are better understood as a result of research on radiation damage and hydrogen pickup, and a partial delineation of the conditions which lead to ignition of these metals in oxygen.

5. Zirconium alloys have been developed which are more resistant to radiation-induced corrosion in uranyl sulphate solution.

6. Progress has been made toward understanding the factors controlling the structure of zirconium and titanium alloys.

FABRICATION OF ZIRCALOY-2 PLATE

Completely isotropic plate is desirable for applications in pressure vessels where tri-axial stresses exist, such as around entrance and exit regions. As would be expected, the anisotropy of Zircaloy-2 † plate can arise both from preferred orientation of the grains and from stringers elongated in the rolling direction.

The microstructure in Fig. 1 shows an intermetallic stringer that can result from conventional fabrication procedures.[1] The inverse pole figure of Fig. 2[2,3] shows the degree of preferred orientation resulting from such a procedure. The intermetallic stringers shown consist of the Fe, Ni, Cr, and Sn intermetallics and are produced in the following way. If Zircaloy-2 is held at a temperature within the lower part of the two-phase field (alpha plus beta), the beta phase will tend to form in the grain corners and will dissolve Fe, Ni, and Cr at the expense of the adjacent alpha material. On rolling, the beta phase will be extended in the rolling direction. On cooling, the intermetallics will precipitate from the elongated beta phase, forming sheets or rods.

The following rolling and heat treating schedule was devised to eliminate the elongated intermetallic stringers and to decrease the degree of preferred orientation.[4] The material was forged between 970 and 1050°C, rolled to within 125% of final plate thickness between 500 and 785°C, beta heat treated and quenched, finally rolled to finish gauge at 480–540°C and then alpha annealed and quenched. Zircaloy-2 plate, sheet, and rod have been successfully fabricated commercially by this schedule.

A typical microstructure for such plate is shown in Fig. 3. Comparison with Fig. 1 reveals the elimination of intermetallic stringers and considerable reduction in preferred orientation which is effected by the new schedule. The stringer appearing in Fig. 3 is a gas stringer, formed from gas entrapment during melting. The effect of beta treatment was shown by rerolling some of the plate previously studied, following the latter part of the recommended schedule, and determining the change in preferred orientation. The dotted lines in Fig. 2 are from this refabricated material and show a 50% reduction in intensity and a 20° rotation of the peaks, indicating reduced anisotropy.

The effect of preferred orientation on ductility can be assessed by the examination of broken tensile

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* Oak Ridge National Laboratory, operated by Union Carbide Corporation for the U.S. Atomic Energy Commission.

† Zircaloy-2 is the designation given to the commercial sponge-base zirconium alloy whose nominal composition is 1.5% Sn, 0.1% Fe, 0.05% Ni, 0.1% Cr and Zr remainder.

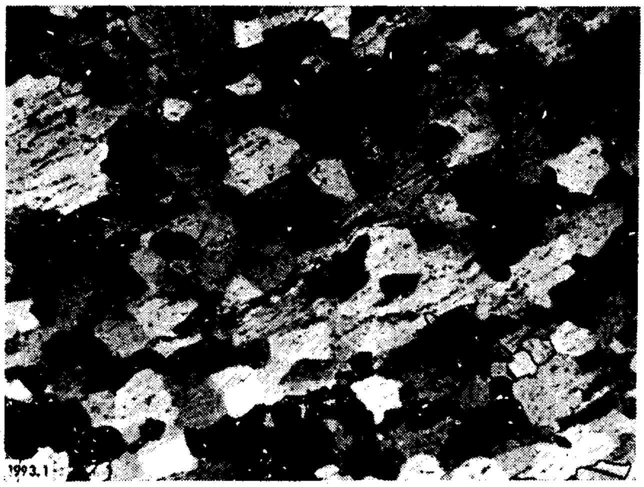

Figure 1. Microstructure of a Zircaloy-2 plate fabricated by conventional commercial practice, showing preferred orientation and large intermetallic stringers: chemically polished, anodized; polarized light. ×350

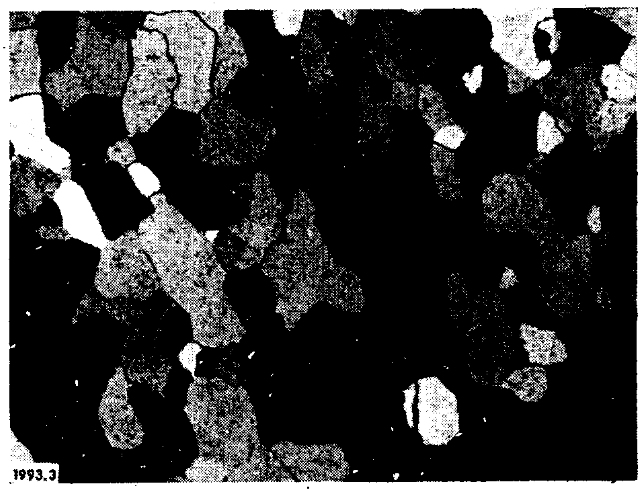

Figure 3. Microstructure of a Zircaloy-2 plate fabricated by developed schedule, showing reduced preferred orientation, absence of intermetallic stringers, but presence of small gas stringers: chemically polished, anodized; polarized light. ×350

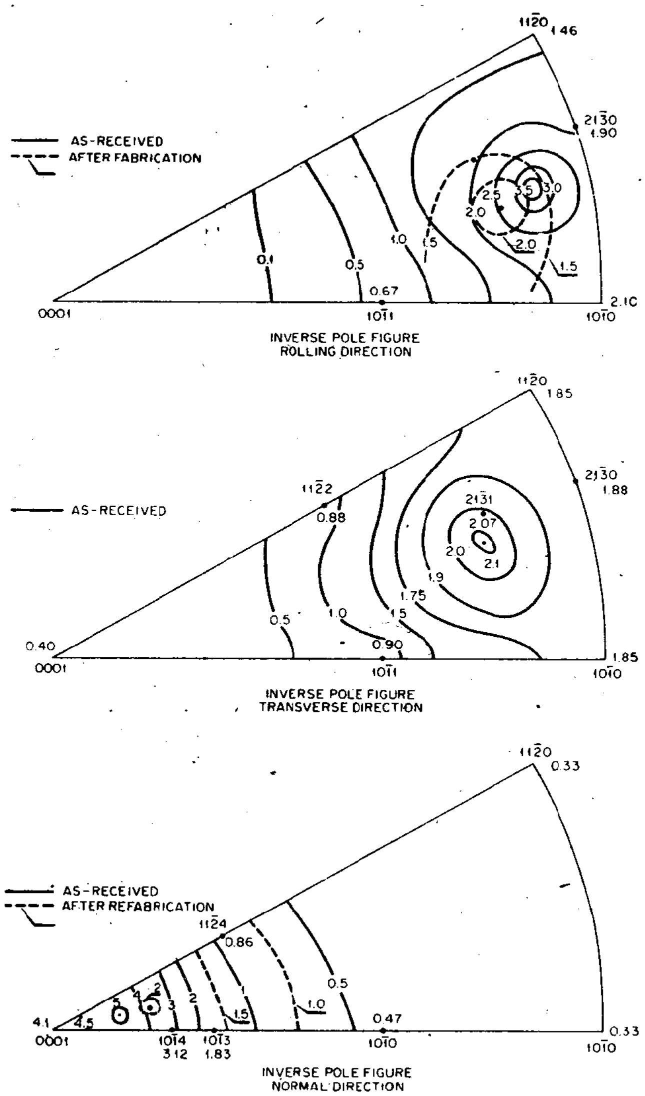

Figure 2. Inverse pole figures showing decrease in anisotropy by developed fabrication schedule: solid lines, as-received; dotted lines, after refabrication

specimens and by Charpy impact tests. In these metals, the cross sections of both the necked portion and fracture surface of tensile specimens are elliptical. For Zircaloy-2 plate, the major axis of the ellipse was always oriented in a direction normal to the plate surface, indicating less ductility in this direction. The lengths of the two axes of these ellipses are reproducible and the ratio between the percentage reduction in the two directions provides a measure of the anisotropy. With the improved fabrication schedules, these ratios have been reduced by a factor of 5 to 6. The reduction in the anisotropy of Zircaloy-2 plates can also be shown by impact tests. Figure 4 presents Charpy V notch impact curves, obtained with samples, and notches, cut from various orientations of a plate fabricated by the conventional schedule and from material fabricated by the improved schedule. These latter curves show more nearly uniform impact energies and transition regions.

WELDING

Large structures would be very difficult to fabricate from zirconium or titanium alloys using inert-atmosphere boxes to provide protection from contamination. Furthermore, the method of shielded welding, which was used to fabricate the Homogeneous Reactor core tank and which was described at the first Geneva Conference,[1,5] is not adaptable for the field welding of complex structures, such as the piping associated with the Homogeneous Reactor. Recently, it has been found possible to make acceptable weldments in unalloyed titanium using only conventional tungsten-electrode, inert-gas, arc-welding equipment.[6] The same procedure can be used somewhat less successfully for welding Zircaloy-2.

In this new procedure, the contamination is reduced by maintaining a complete, inert-gas blanket and increasing the freezing rate of the deposited metal. The face of the weld is blanketed without turbulence

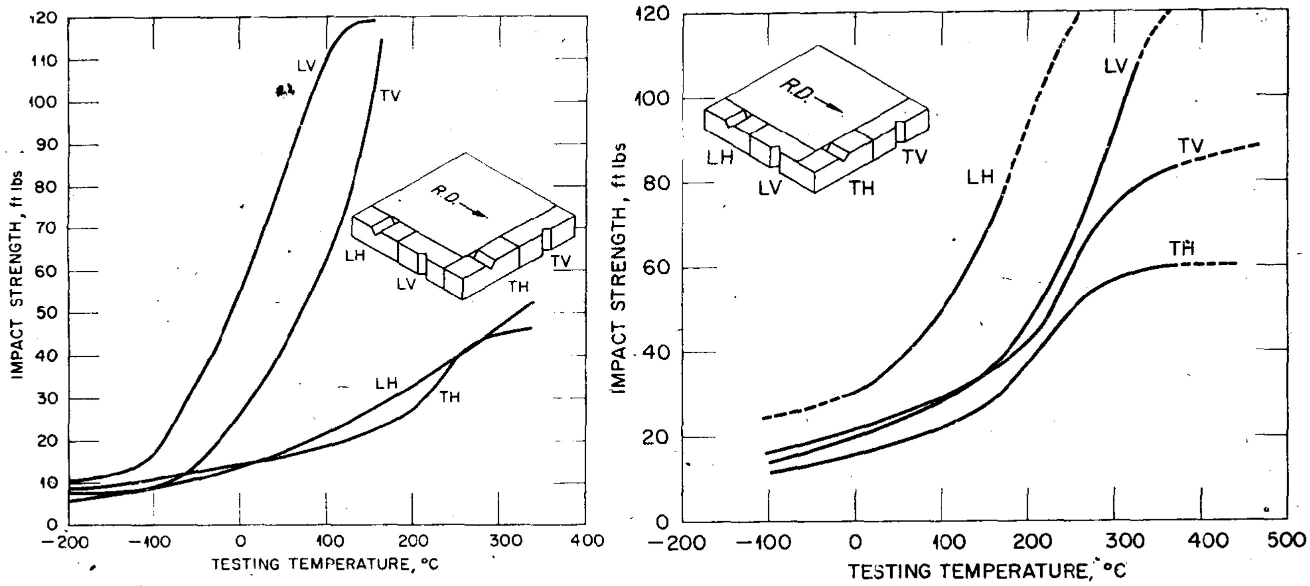

Figure 4. Charpy V impact energy curves for various specimen orientations of Zircaloy-2 fabricated by: left, conventional commercial schedules; right, by developed schedule

by carefully controlling the gas flow rate and by using a large gas cup. The root of the weld is protected by inert gas back-up of the weld zone. A smaller molten pool is produced by reducing the power input and by using small root and land spacings, thus reducing the area and time available for contamination to occur.

It was found that such welds would have adequate toughness if the hardness did not exceed 220 DPH. This requirement could be met consistently in unalloyed Ti weldments, but, at present, it is not possible to keep the hardness of Zircaloy-2 weldments below 220 DPH. Nevertheless, a considerable degree of toughness is maintained in Zircaloy-2 welds.

HAZARDS

Three factors of importance to the safety of reactors using zirconium and titanium alloys are the effect of radiation on their mechanical properties, the possibility of hydrogen pickup and embrittlement, and the effect of operating conditions on their tendency to react with oxygen.

Subsize tensile and impact specimens of Zircaloy-2, crystal bar zirconium, and A40 titanium have been irradiated in fissioning aqueous uranyl sulfate solutions at temperatures of 250 to 280°C and to integrated fast fluxes (> 1 Mev) of up to 3×10^{19} nvt for zirconium and 10^{19} nvt for titanium. The latter undergoes slight embrittlement with increases in tensile and yield strengths and decreases in reduction of area amounting to about 10% of the values of unirradiated material. On the other hand, zirconium appears not to suffer from embrittlement, since only in the yield point were changes noted, these being very small.

In an aqueous reactor, hydrogen may be formed by both corrosion reactions and by dissociation of the water. If such hydrogen should be picked up by the zirconium, severe embrittlement would result. Samples of Zircaloy-2 and crystal bar zirconium have been placed in the in-pile corrosion loops and exposed to fissioning uranyl sulfate at 250° to 280°C for more than 1000 hr. Under the heavily oxidizing conditions of these loops, very little, if any, hydrogen was picked up by the zirconium.

Unfortunately, both massive Ti and Zr can react quite rapidly with oxygen under circumstances where the ordinary engineering alloys are quite inert. The conditions under which such reactions occur have been investigated by E. M. Kinderman[7] and coworkers of Stanford Research Institute.

Two types of tests were developed for studying the ignition reactions; in one, a dynamic-atmosphere test, a thin metal disk was fractured by either gas pressure or by a plunger; while in the other, a static-atmosphere test, either a thin sheet or a $\frac{1}{4}$-in. rod was broken in tension. In either test, composition, pressure, and temperature of the atmosphere could be varied. It proved to be surprisingly easy to initiate combustion of titanium in both types of tests. Ignition and complete consumption of both disks and rods occurred when titanium and Zircaloy-2 were ruptured even at room temperature in a high pressure of pure oxygen.

The limiting conditions for ignition of A55 titanium are shown in Fig. 5. The upper curve was determined under the static conditions of the tensile-type test, while the lower curve was determined using a disk ruptured mechanically and with high velocity gas passing through the rupture. Reactions occurred under conditions shown by the area above the lines but not under those below.

In a high-velocity stream of pure oxygen, reaction occurred at pressures as low as 50 psi with titanium. The lowest curve in both figures becomes asymptotic

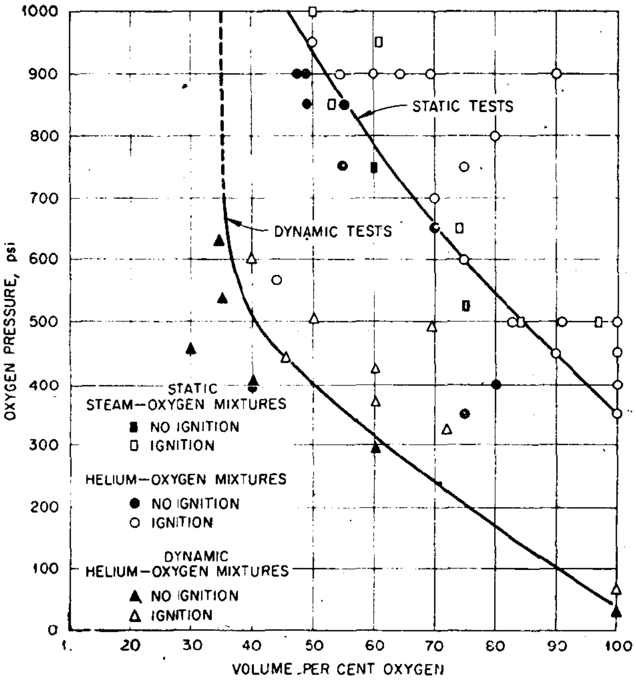

Figure 5. Limiting conditions for titanium combustion reactions according to tests at the Stanford Research Institute

with the pressure axis at about 35% oxygen, indicating that no reaction will take place with leaner mixtures at any pressure. It should be pointed out that, with higher velocities than those studied, it is possible that the critical oxygen pressure is even lower than shown.

A point of major interest in the consideration of the use of titanium in aqueous reactors, with their steam pressurization, is that similar curves result when the oxygen is diluted with either helium or steam. The water vapor apparently does not enter into the ignition reactions. As would be predicted from these curves no reaction takes place when the sample is fractured under water even if the water is saturated with oxygen.

While zirconium is similar to titanium in that auto-ignition can occur, the critical oxygen pressures appear to be considerably higher. Where under dynamic conditions titanium ignited with 50 psi oxygen, a zirconium disk required 500 psi. Under static conditions a 0.015-in. thick strip of titanium ignited at a pressure of 350 psi but a similar strip of zirconium required 750 psi. A $\frac{1}{4}$-in. titanium rod ignited under the same conditions as the strip, but a zirconium rod did not ignite at 1500 psi oxygen. On the other hand stainless steel, aluminum, magnesium, iron, tantalum, niobium, and molybdenum did not ignite at oxygen pressures as high as 2000 psi.

Although the pressures of the present reactor concepts do not fall within the reaction range, the ignition hazard must be considered in future designs.

In summary: neither titanium nor zirconium appears to be very susceptible to radiation damage; both titanium and zirconium can react rapidly if fractured in an oxygen-enriched gas, but fortunately not in oxygenated water; and hydrogen pickup of Zr and Ti alloys does not appear, at the present time, to be a hazard in oxidizing environments.

ZIRCONIUM ALLOY DEVELOPMENT

Zircaloy-2 is satisfactorily resistant to corrosion by uranyl sulfate solutions in the absence of radiation. However, if the metal–liquid interface is subjected to radiation by heavy fission fragments, the corrosion rate increases sharply. It has been found that this rate can be reduced by alloying zirconium with Nb, Pd, or Pt. The zirconium–niobium-base alloys can be heat-treated to attain high strengths with moderate ductilities. However, under many conditions, brittle alloys result, creating problems in welding. Sufficient metallurgical data have not been obtained on the Pd or Pt binary systems to include a discussion of them in this report.

The hardness of Zr–15% Nb alloys, quenched from the beta region and aged, increases with time at a variety of temperatures as shown in Fig. 6. It is clear that hard and presumably brittle structures are to be expected in multipass welds in such alloys. Studies are being made to delay such hardening by the addition of ternary solutes. That success in this direction has been achieved by the addition of 2% Pd or 2% Mo to the 15% Nb alloy can be seen by comparing Figs. 7 and 8 with Fig. 6.[8]

Metallographic and X-ray diffraction studies are being made of the structural changes accompanying the hardness changes. It has been found that considerable differences in hardness can occur without visible changes in microstructure, as can be seen in Fig. 9.

It appears that there are at least three metastable transformation products in the Zr–15% Nb alloy and that the transformation sequence is quite complex. It has been demonstrated, however, that the major hardening is produced by the decomposition of the retained beta phase to omega plus beta enriched in Nb. The omega-phase structure has been found to be hexagonal, in agreement with the work of Silcox, Davies, and Hardy[9] on Ti–V alloys.‡

Cursory fabrication studies have been performed during the course of the alloy development program in the preparation of sheet specimens for the transformation kinetics study.[13] All of the Zr–Nb–X alloys have been hot-rolled from 800°C quite successfully. A sponge-base Zr–15% Nb arc-casting has been successfully extruded at 950°C.

A small amount of mechanical property evaluation of the Zr–15% Nb alloy has been performed. An

‡ If this structure be heated to 600°C, the omega phase disappears within two minutes. At the end of two hours, the structure consists of alpha plus enriched beta. At the end of two weeks it consists of alpha, enriched beta, and the niobium-rich phase. These results are consistent with the Zr–Nb diagram as determined by Rogers and Atkins.[10] The dilatometric arrests found by Bychkov, Rozarrov, and Skorov,[11, 12] at 550°C are presumably to be attributed to the changes outlined above and not to a eutectoid transformation.

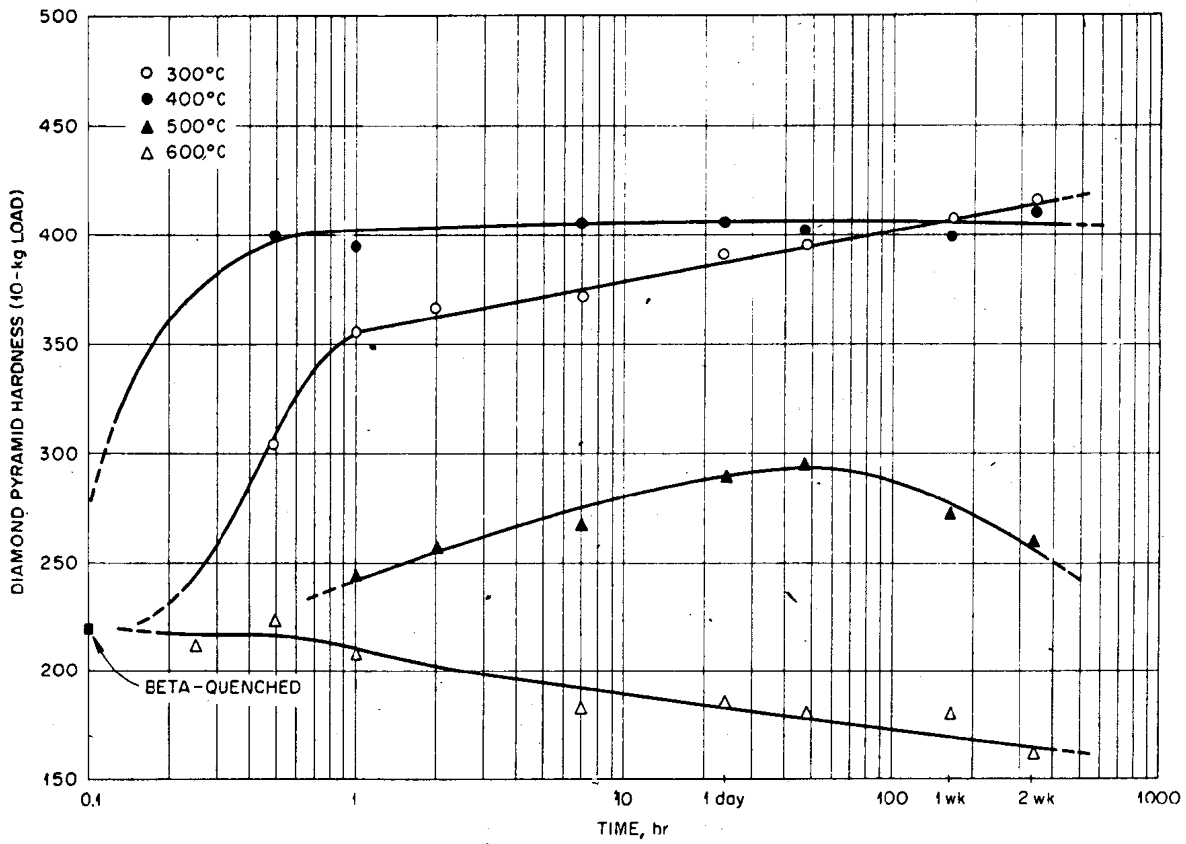

Figure 6. Time, temperature hardness curves for beta-quenched and reheated Zr-15% Nb alloys

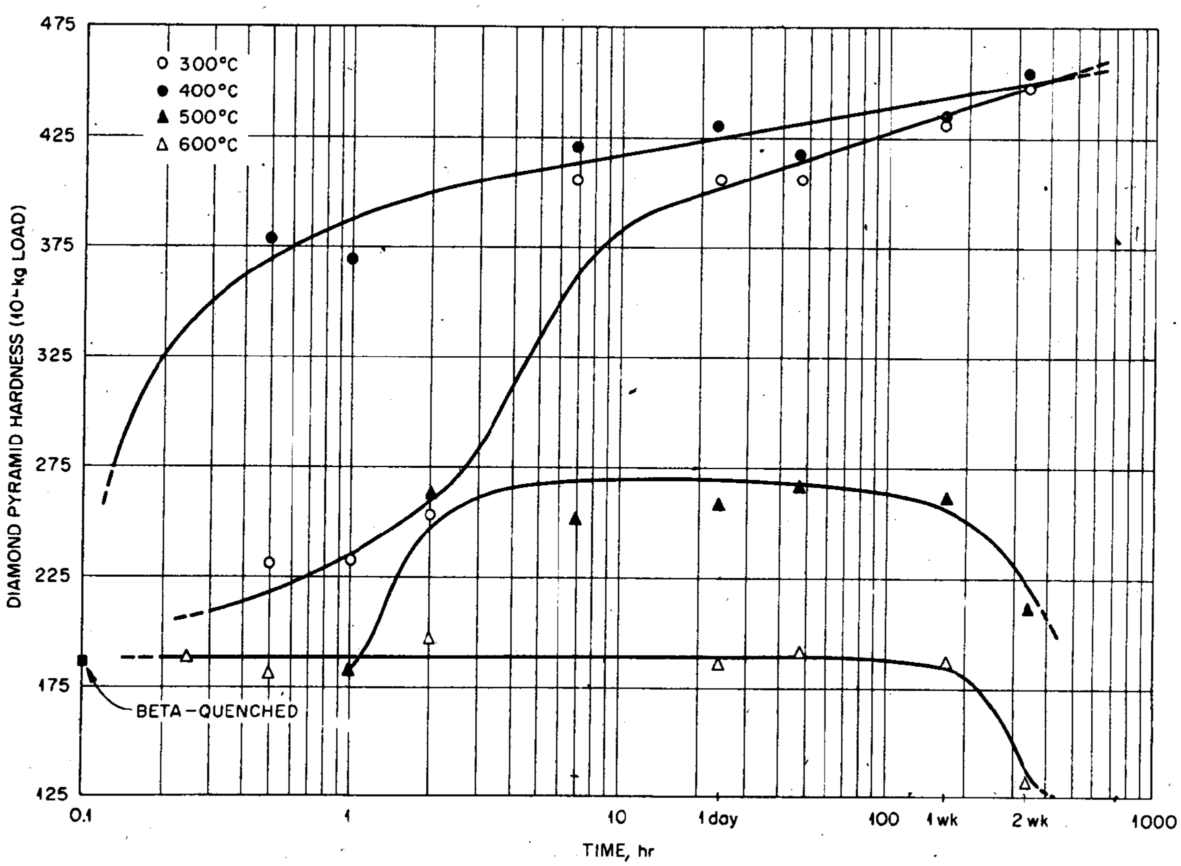

Figure 7. Time, temperature hardness curves for beta-quenched and reheated Zr-15% Nb-2% Pd alloys

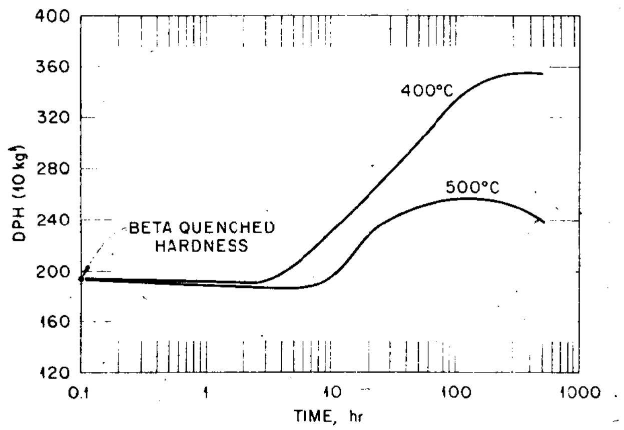

Figure 8. Time, temperature hardness curves for beta-quenched and reheated Zr–15% Nb–2% Mo alloys

ultimate tensile strength, at room temperature, of 200,000 psi (with no elongation), was obtained on specimens beta-quenched and aged at 400°C for two hours. An ultimate strength of 150,000 psi, a yield strength of 135,000 psi, and an elongation of 10% at room temperature has been obtained for a Zr–15% Nb specimen aged at 500°C for two weeks. The same aging produced an ultimate strength of 105,000 psi, a yield of 90,000 psi, and an elongation of 16% in one inch at 300°C.

While the Zr–15% Nb-base ternary alloys are still of interest primarily for their potential improvement in corrosion resistance, they show promise of a wider, more general use. These alloys are the first zirconium alloys that may be treated to high strength and yet are weldable and fabricable alloys. They show promise of becoming a structural material usable in reactors.

FUNDAMENTALS OF ALLOY BEHAVIOR

In addition to the development of new alloys, progress has been made in understanding zirconium and titanium alloys from a fundamental standpoint. The starting point in this work has been an examination of the binary equilibrium diagrams.

As a result of the electropositive natures of zirconium and titanium, the more concentrated alloys are nearly always characterized by brittle, intermediate phases of narrow solubility range. These phases are especially stable with electronegative solutes like Si, Ge, Sb, B, C, N, and O, or with Group VIII solutes such as Co, Ni, and Pt. On the other hand, they are much less stable with elements such as Cu, Ag, and Au, and with other elements of Group IV-A they are absent entirely. These intermediate phases are of considerable theoretical interest, but, for reactor construction, the region of interest is limited to the zirconium-rich and titanium-rich alpha and beta phases.

Understanding of the factors governing the phase boundaries between these terminal phases has been possible in terms of two factors: the electron concentration, and the differences in the sizes of the atoms. A detailed discussion of this equilibrium has been given recently by Betterton and Frye.[14] The relationships are illustrated, for Zr, in the partial phase diagrams of Fig. 10 arranged according to the position of the solute in the Periodic Table. This figure also includes recent work on Zr with Ag, Cd, In, Pb, and Sb at the Oak Ridge National Laboratory[15–19] and on Zr–Pt by Kendall[20] at the University of Kentucky. Titanium-base alloy systems have also been plotted in the same way and the effects observed are similar to those for zirconium.

In both Zr and Ti alloys, the α/β phase boundaries are depressed and a eutectoid-type system results with each of the transition elements and with the elements Cu and Ag. The α/β boundaries are raised by elements from B subgroups, III-B, IV-B, and V-B. This correlation is improved by making an allowance for atomic size effects which, when large, rotate the boundaries downwards. With appropriate allowance for size effects, a linear relationship between the electron concentration and the mean temperature of the α/β boundaries can be deduced in terms of the conventional metallic valencies for the solutes as follows: 1 for Cu and Ag, 2 for Cd and Zn, 3 for Al and In, 4 for Sn and Pb, 5 for Sb, and 0 to 1 for transition elements. This relationship is consistent when Zr and Ti are made equivalent in valency to Cd, or divalent in the metallic state. The divalency implies that two of the four outermost electrons, presumably the 4d electrons of the free atom, are not important in alloy formation. Such a situation would make alpha Zr and Ti analogous to hexagonal Mg where the Brillouin zone structure is fairly well understood and where the addition of electrons by alloying expands the axial ratio. In fact, this effect does occur in Zr[21] and Ti[22] although a complete understanding of the alloying effects of the axial ratio is not currently available; in particular, because there are uncertainties about the role of the d electrons in this process. Investigations of these alloys are continuing with an investigation of the electronic specific heat at liquid helium temperatures. The initial results for the alpha phase of the Zr–In system[23] show an increase in the density of states of Zr, which is large enough to measure accurately and which agrees with the expansion of the axial ratio by In alloying in terms of a Brillouin zone overlap. When these measurements have been completed, with solutes of other valencies, more definite information will be available about the validity of various theoretical models for these Ti and Zr alloys.

As can be seen in Fig. 10, the elements which permit choice of alpha- or beta-type alloys are clearly defined by this electron concentration and size-factor rule. The possibilities of mechanical-property improvement by precipitation processes or the avoidance of the allotropic transformation in fabrication or thermal cycling can thereby be predicted in these alloys.

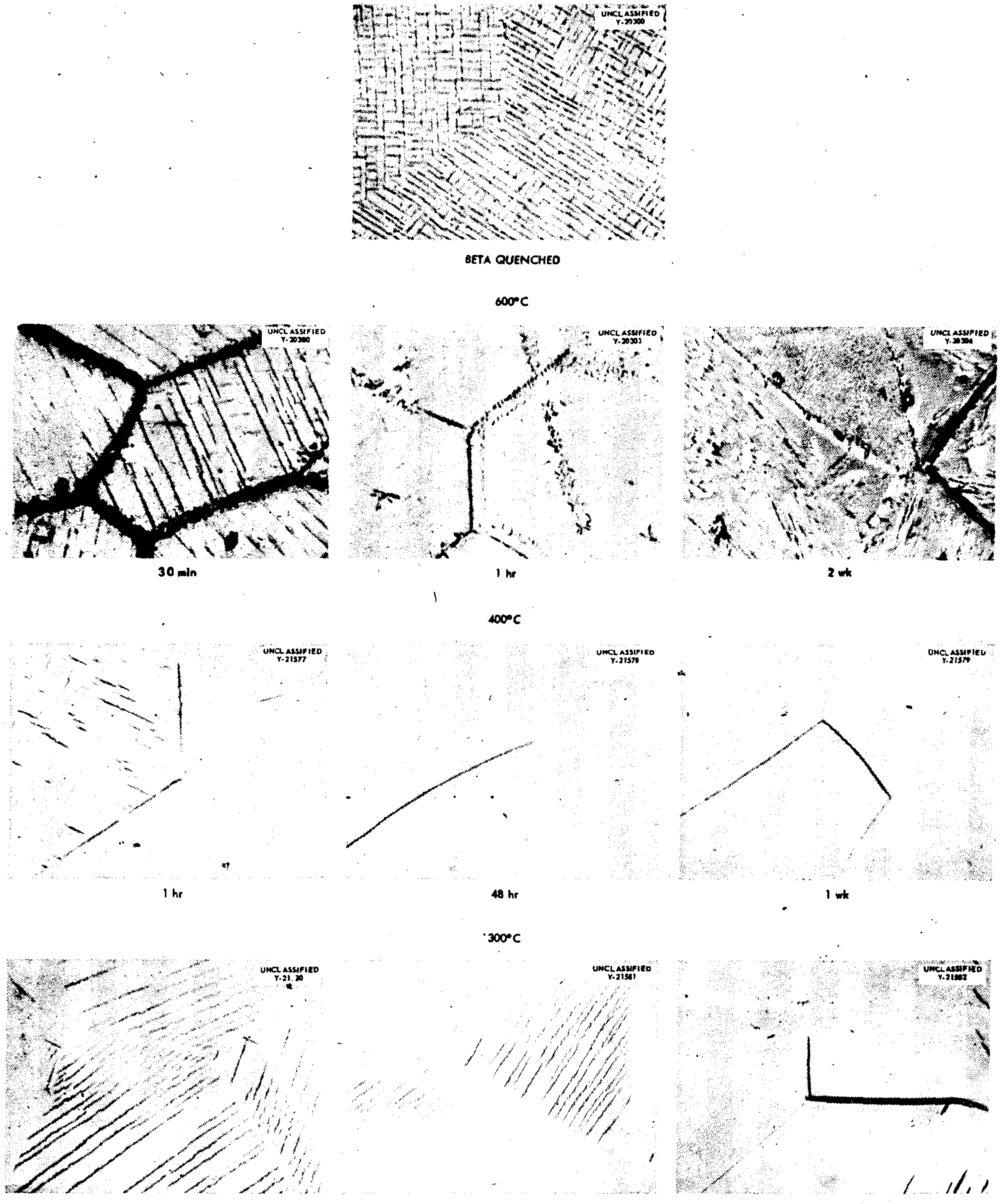

Figure 9. Microstructures of beta-quenched and aged Zr–15% Nb alloys

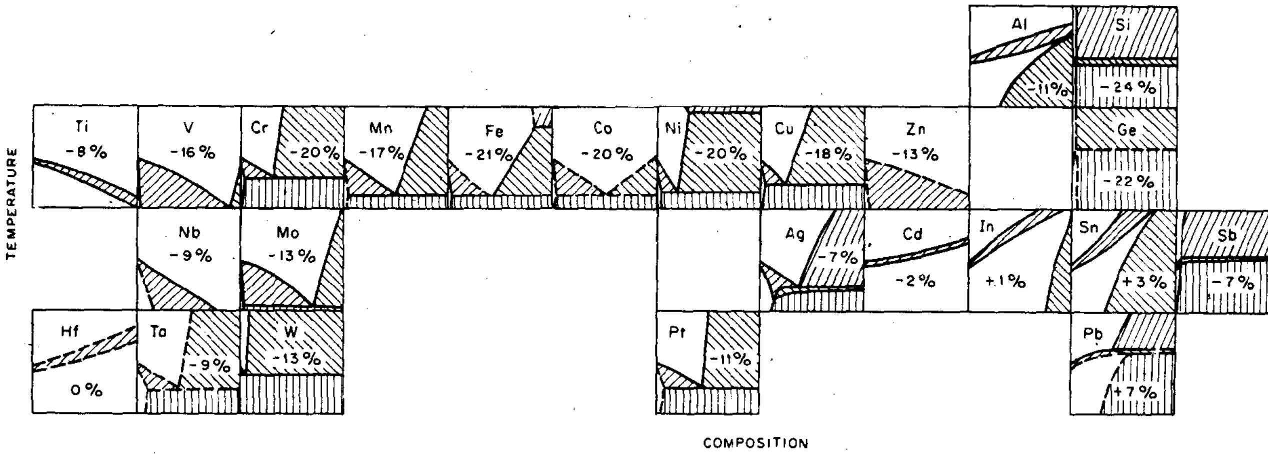

Figure 10. Binary Zr phase diagrams between the compositions of 100–90 Zr and temperatures 775–975°C Numbers represent atomic size differences

CONCLUSION

In summary, Zr alloys are currently available with better neutron and corrosion properties than any other known reactor material of comparable strength and ductility. Progress has been made towards resolving some of the special problems in these and certain Ti alloys such as fabrication and welding procedures, improved corrosion resistance under heavy particle bombardment, and the delineation of burning hazards. Ultimately, alloy development may permit full utilization of the strong binding forces in these metals, indicated by their high melting point and heat of vaporization, in terms of mechanical strength at higher temperatures. More immediately, alloys which are less sensitive and more resistant to contamination in fabrication are urgently required to reduce the cost of preparing zirconium and titanium materials.

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