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SUBJECT Report on Experiment
LSR-a6, "Hot Malleability
of Zircaloy-2 and High
Zirconium-Uranium Alloys"

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Attached is a report on Experiment LSR-a6, "Hot Malleability
of Zircaloy-2 and High Zirconium-Uranium Alloys".

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HOT MALLEABILITY OF ZIRCALOY-2
AND HIGH ZIRCONIUM-URANIUM ALLOYS

SUMMARY

One-inch cube and one-inch cylindrical specimens of Zircaloy-2 and the 50, 60, 70 and 80 w/o zirconium-uranium alloys have been hot compression tested at 1600, 1500, 1350, 1250, 1150 and 1000°F. The technique is an adaptation of a hot malleability test used on nickel alloys by the International Nickel Company. It was found that Zircaloy-2 showed poor malleability at certain temperatures by edge cracking in the direction of upset, a type of failure very similar to that found in nickel alloys. The data indicate that Zircaloy-2 has good malleability as low as 1500°F at all reductions, and at temperatures as low as 1000°F if reductions are limited to 50 percent.

The zirconium-uranium alloys showed good malleability at all temperatures except 1000°F, where specimens failed in shear. It is felt that this type of hot compression test needs some refinement as to method and interpretation of results when used on zirconium-uranium alloys, since limited fabrication results indicate that there is a difference in rolling and forging characteristics at these temperatures which are not brought out by hot malleability results. Data did reveal that greater reductions could be taken resulting in more uniform metal flow and a greater yield.

INTRODUCTION

One of the problems of fuel element development is the determination of the fabricability of various fuel alloys. This is always an expensive process since it involves the forging and rolling of relatively large amounts of material to evaluate the effect of temperature and the amount of reduction between heating

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times. The process is expensive in time as well as money because the material being evaluated is usually earmarked for other experiments and initial attempts at fabrication are not always successful. To limit the number of variables, the practice has been to keep the amount of reduction constant for all alloys and simply determine the effect of temperature. Reductions have been limited to 1/4" per pass since many uranium alloys have tended to surface crack with greater reductions. This limits the initial reduction on a 4" diameter ingot to less than 6 percent, which is not sufficient to cause metal flow near the center of the ingot.

It was hoped that a hot malleability test of some sort might aid in predicting what to expect during forging and rolling operations.

The International Nickel Company* has been using hot malleability tests since 1930. Up to 1948, 63,000 commercial melts of Monel, nickel, Inconel and other high nickel alloys have been tested and the test data compared with processing results. Two methods have been standardized on; the hot 180° bend test and the hot compression test. The preparation of test specimens consists of pouring a 2" diameter ingot directly from the teeming ladle after the first commercial size ingot of a melt is poured. The ingot, without prior overhauling, is hammer forged at an optimum forging temperature to 1" x 1" and 1/2" x 1" rod. One-inch cube specimens are sawed from the first rod for hot compression testing. Four-inch lengths are taken from the second for the 180° bend test. The hot compression test consists of placing a 1" cube specimen that had been heated to the

*"Methods of Evaluating Hot Malleability of Ni and Hi-Ni Alloys"; L. H. Martin and L. O. Bieber; Nonferrous Rolling Practice, Institute of Metals Division Symposium Series, Volume 2, AIME, New York, 1948, page 15.

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testing temperature on the bottom die of a steam hammer with one of the prior forged surfaces resting on the die, and quickly compressing it with one blow. Compression tests are repeated on other 1" cubes increasing the reduction in thickness 1/16" at a time until the maximum amount which a specimen can be compressed without fracture is determined. Two types of fractures normally occur. The first type is irregular, runs in the direction of compression and may occur on all four side surfaces. The second type runs diagonal to the direction of compression and occurs only on the two side surfaces representing the cross-section of the forged 1" square rod. The first is of principal interest and is an indication that the metal will tend to surface or edge crack when worked at the testing temperature. The second type is a function of hardness and structure.

The 180° hot bend test consists of heating the specimen to the test temperature and bending the specimen over on itself. Evaluation is simply a matter of observing whether or not fracture occurs on the outside bend surface.

The hot compression test has the advantage that results can be reported as the limit of reduction at any one temperature before side cracking occurs. However, a number of specimens are required to evaluate each temperature. The 180° bend test has the advantage that only one specimen is needed to test a temperature. The amount of reduction is related to deformation of the outside surface during bending and has been found to be equivalent to approximately 43 percent compression. Thus, malleability at reductions higher than 43 percent cannot be evaluated with the bend test.

The reason given for using forged rather than cast specimens is principally that the grain structure of a commercial size ingot cannot be reproduced in a small cast test ingot. Both types of specimens will show the type of fracture caused by the inherent lack of malleability of the metal at the temperature at

which it is hot worked; however, cast specimens will also sometimes show rupture which is associated with the cast grain structure of the ingot. This second type of rupture cannot be accurately predicted by the hot compression test. Thus, cast specimens tend to show erratic behavior and are therefore not as useful as forged specimens for determining hot malleability.

In this report, the hot malleability of Zircaloy-2 and the 50, 60, 70 and 80 w/o zirconium-uranium alloys is discussed. The hot compression test, instead of the 180° bend test, was selected because of its greater flexibility in spite of its greater material requirements. Hot malleability data for these alloys are compared with limited fabrication results at WAPD and with the extensive experience obtained by the International Nickel Company on nickel alloys.

PROCEDURE

Hot malleability specimens used in this experiment were of two different shapes. The Zircaloy-2 and 60 and 70 w/o zirconium-uranium specimens were forged at 1600°F from 4" diameter ingots to 1-1/4" diameter octagons, from which 1" diameter x 1" right circular cylinders were machined. The 50 and 80 w/o zirconium-uranium specimens were made by sawing 1" lengths from as-forged 1" square rod, the forging temperature being the same as that above. The testing procedure was to place either the flat surface of the cylindrical specimens or one of the as-forged surfaces of the cube specimens, after heating to the testing temperature, on the 300-ton press anvil and upset in one blow to one of the 8 thicknesses varying from 7/8" to 1/8", representing reductions in thickness from 12 to 88 percent. Thus, the machined cylindrical specimens were compressed in the direction of forging, whereas the as-forged cube specimens were tested perpendicular to the direction of forging. Testing temperatures were so chosen that those above and below transformation and solvus lines as shown by the binary equilibrium

diagram would be represented and also so that a comparison could be made between alloys. The temperatures tested were 1600, 1500, 1350, 1250, 1150 and 1000°F. The relationship between testing temperature and composition is shown on the binary equilibrium diagram for the uranium-zirconium system, Figure 1. Specimen heating was done in a carbonate* salt bath for 3 to 3.5 minutes at temperatures above 1000°F and for 10 minutes at 1000°F.

Specimens that had been upset to a 12 and 50 percent reduction in thickness were sawed in half perpendicular to the direction of upset and machined for measurement of Rockwell "C" hardness. Those upset to either an 88 or 75 percent reduction in thickness were sawed in the direction of upset. In all cases, the sawed surface was machined prior to hardness measurement.

RESULTS

Compression testing of Zircaloy-2 and 50, 60, 70 and 80 w/o zirconium-uranium specimens according to the above procedure shows good malleability at temperatures at and above 1150°F. At 1000°F all of the zirconium-uranium alloys appear to fail brittly at all reductions, as noted in Table I. In contrast, Zircaloy-2 was the only alloy tested that failed by irregular fracture in the direction of compression, indicating hot shortness of the type that also occurs in nickel alloys. The brittle nature of the failure of zirconium-uranium specimens when upset at 1000°F as contrasted with excellent hot malleability at 1150°F is illustrated in Figures 2 and 3. Table I under the heading "Edge Cracking" shows that none of the zirconium-uranium specimens failed by edge cracking. In contrast, it can be seen from the table that Zircaloy-2 has poor malleability between the temperatures of 1000 to 1350°F, as evidenced by edge cracking of hot compression

*Houghton's Liquid Heat 235 - 28% Li_2CO_3 , 33% Na_2CO_3 , 39% K_2CO_3

specimens, when reductions in thickness are over 50 percent. The bottom specimen in the Zircaloy-2 pile of specimens in Figure 2 is an excellent example of edge cracking.

The peripheral surface of the machined cylindrical Zircaloy-2 specimens were very smooth after upsetting, whereas that of the zirconium-uranium alloys were rippled, with the ripples running in the direction of compression. This phenomenon is illustrated in Figures 2 and 4. A comparison of Figures 2 and 3 shows that the rippling of the zirconium-uranium alloys is much less pronounced in the cube specimens.

Specimens which had undergone a relatively large upset invariably surface cracked radially on surfaces in contact with the anvils. This surface checking was always worse on the surface of the specimen which was in contact with the bottom die of the press. Figure 5 is a comparison of the degree of anvil surface cracking as a function of temperatures for the 80 w/o zirconium-uranium alloy upset to a 75 percent reduction in thickness. The surface that had been in contact with the bottom anvil is turned up in each case. It is obvious from the figure that anvil-surface cracking decreases to a minimum somewhere near 1150°F for this alloy. Generally, the cylindrical specimens show a greater amount of surface checking than the cube specimens.

Some zirconium-uranium specimens upset to an 88 percent reduction in thickness split within one day due to internal stresses. Within a month and a half, additional specimens that had been upset 75 percent had cracked or split open. Figure 6 serves to illustrate the two typical types of fractures that did occur. Table I also indicates which specimens cracked or broke due to internal stress and shows that the susceptibility to cracking seems to decrease with decreasing uranium in the alloy.

Closely akin to cracking due to internal stress is specimen breakage during machining for hardness measurement. However, in contrast to breakage due to internal stress, breakage during machining seemed to increase with decreasing uranium content. The two effects can be compared in Table I.

Figure 7 is a plot of the recorded hardness. Each point represents five readings on one specimen.

DISCUSSION OF RESULTS

The present practice of press forging uranium alloys has been to limit the reduction to only $1/4$ " per pass because of the general susceptibility of such alloys to surface cracking or tearing. The lack of edge cracking at all reductions on the zirconium-uranium alloys when compression tested between 1150 and 1600°F is evidence of good malleability. This suggests that reductions greater than $1/4$ " per pass might be attempted on $1/2$ " diameter ingots in order to obtain a greater amount of metal flow in the center of the ingot, which should result in a more uniformly worked structure and a greater yield. The severe anvil surface cracking, however, is evidence that good malleability may be precluded. From past experience, temperatures of 1500 to 1600°F have been used with reasonably good results. An increase in reductions per pass to $1/2$ " has shown no deleterious effects on the 50 w/o alloy, although the 80 w/o zirconium-uranium alloy still exhibits some surface cracking. A $1/2$ " diameter 60 w/o zirconium-uranium ingot forged very well at 1250°F to a 2" x 2" bar, taking $1/2$ " reductions per pass. However, once this size had been reached, the 75-ton capacity press could not enforce greater than $1/8$ " reductions. This caused considerable surface cracking. A $1/2$ hour anneal at 1500°F did not soften the alloy and forging had to be completed at 1500°F. In contrast, the 50, 60, 70 and 80 w/o zirconium-uranium alloys have been successfully

rolled from 1" to 0.1" or less at 1250°F. Temperatures of 1500 and 1600°F cause severe surface cracking when these alloys are rolled thinner than about 1/4".

In view of the apparent conflicting rolling and forging results based on limited evidence, it is difficult to correlate experience with malleability data. It is apparent from the data that failure in the zirconium-uranium alloys occurred in a brittle manner and, therefore, in a different manner than in the nickel base alloys tested by the International Nickel Company. Zircaloy-2, however, exhibited edge tearing in the direction of compression in the same manner as the nickel alloys, making it considerably easier to compare malleability results with known fabrication results. Thus, it might be expected that Zircaloy-2 could be readily forged at temperatures as low as 1500°F. Surface cracking would be expected at temperatures down to 1000°F if reductions exceeded 50 percent.

The zirconium-uranium alloys exhibited a rippling on the side surfaces of hot compression specimens after upset. Rippling was less apparent on specimens that had been upset in the direction of prior forging than those upset perpendicular to this direction. In contrast, Zircaloy-2 exhibited a perfectly smooth side surface.

The data show that some specimens broke from internal stress and that breakage was a function of time. It is known that cooling rates intermediate between a very rapid water quench on small specimens and a very slow cooling rate render these alloys very brittle. The most obvious explanation for breakage seems to be one incorporating hardening by an anvil quench at an intermediate rate coupled with a certain amount of cold working and aging at room temperature. It is surprising to note (Table I) that breakage during machining seemed to increase with increasing zirconium content, whereas, the opposite appeared to be true for breakage attributed to internal stress. This phenomena was considered beyond the scope of this report and, therefore, was not investigated further.

Figure 7 is a graph of the Rockwell "C" hardness measurements made on specimens upset to a 12, 50 and 75 or 88 percent reduction. The Zircaloy-2 and the 60 and 70 w/o zirconium-uranium specimens were upset perpendicular to the direction of prior forging whereas the 50 and 80 w/o zirconium-uranium specimens were upset in the direction of forging. Hardness measurements were made on the center and in the direction of upset on specimens that had undergone a greater reduction. Only the broadest generalizations can be drawn from the data because each point represents an average of five readings on only one specimen. The length of time specimens remained on the anvil before upset varied by about 1 or 2 seconds, and hence, hardness would be expected to vary if the quenching rate happened to be critical. It is apparent from the figure that a very rapid quench, as evidenced by upset to an 88 percent reduction, softens the 50 w/o zirconium-uranium alloy and increases the hardness of the other three zirconium-uranium alloys. In actual practice, hardness of forged and rolled material would be somewhat similar to malleability specimens upset to a 12 percent reduction. Thus, hardnesses of about 35 Rockwell "C" or higher would be expected on as-forged or rolled material of all four alloys.

Zircaloy-2 shows a slight increase in hardness with decreasing forging temperatures.

CONCLUSIONS

Hot compression specimens of the 50, 60, 70 and 80 w/o zirconium-uranium alloys showed no edge tearing in the direction of upset at reductions of from 12 to 88 percent in thickness at 1600, 1500, 1350, 1250 and 1150°F, indicating good malleability at these temperatures. All four alloys failed in shear at all reductions at 1000°F. Limited fabrication results based on this data indicate that greater reductions per pass can be taken without deleterious results. Not much forging data is available at temperatures below 1500 and 1600°F. Aside from this

data, experience indicates that 1250°F is the best rolling temperature to avoid surface cracking, which occurs at 1500 and 1600°F when thicknesses become less than 1/4".

Thus, hot compression testing of these four zirconium-uranium alloys has not foretold whether surface cracking would or would not occur during forging and rolling. It did indicate, however, that these alloys do behave differently than the nickel alloys tested by the International Nickel Company and, hence, would require considerable additional work for adequate interpretation of results.

Zircaloy-2 exhibited edge tearing in the direction of upset similar to the nickel alloys. Interpretation of hot compression data shows that Zircaloy-2 has good malleability at temperatures as low as 1500°F and that surface cracking at temperatures down to 1000°F can be avoided if reductions do not exceed 50 percent. No systematic checking of this data against actual forging and rolling results has been done or is contemplated.

TABLE 1 SUMMARY OF GENERAL EFFECTS ON HOT MALLEABILITY SPECIMENS

COMPOSITION	TYPE OF SPECIMEN	PERCENT UPSET	EDGE CRACKING	GENERAL CRACKING						SURFACE CHECKING						BROKE DUE TO INTERNAL STRESS WITHIN 1 DAY								
			UPSET TEMPERATURE (°F)																					
			1600	1500	1350	1250	1150	1000	1600	1500	1350	1250	1150	1000	1600	1500	1350	1250	1150	1000				
50 w% Zr-U	FORGED CUBE	12					*						*					*						*
		25						*					*					*					*	
		37						*					*					*					*	
		50						*											*				*	
		62						*					*						*				*	
		75						*					*						*				*	
		88						*					*						*					*
60 w% Zr-U	MACHINED CYLINDER	12																						
		25																						
		37																						
		50																						
		62																						
		75																						
		88																						
70 w% Zr-U	MACHINED CYLINDER	12																						
		25																						
		37																						
		50																						
		62																						
		75																						
		88																						
80 w% Zr-U	FORGED CUBE	12			*						*					*			*					*
		25			*						*					*			*				*	
		37			*						*					*			*				*	
		50			*						*					*			*				*	
		62			*						*							*				*		
		75			*						*							*				*		
		88			*						*							*				*		
Zr 2	MACHINED CYLINDER	12																						
		25																						
		37																						
		50																						
		62																						
		75																						
		88																						

* SPECIMENS NOT TESTED

Figure 1

Zirconium-Uranium Constitution Diagram

Figure 2

M 1469-3

A comparison of cylindrical specimens upset at different temperatures. From left to right:

- (a) 70 w/o Zr-U upset at 1150°F showing no edge cracking at any reduction.
- (b) 70 w/o Zr-U upset at 1000°F showing brittle failure at almost all reductions.
- (c) Zr-2 upset at 1000°F clearly showing edge cracking at the greatest reduction.

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Figure 3

M 1472-2

A comparison of the 80 w/o Zr-U alloy upset at 1150°F (left)
and 1000°F (right).

Figure 4

M 1469-2

Comparison of the two shapes of specimens used and the difference in edge surface appearance between Zr-2 and a Zr-U alloy in the upset cylindrical specimens. From left to right: Zr-2, 60 w/o Zr-U and 50 w/o Zr-U, all upset at 1500°F to a 50 per cent reduction in thickness.

Figure 5

M 1472-1

A comparison of the surface of the 80 w/o Zr-U alloy upset to 75 per cent reduction in thickness at (from left to right, top row first) 1600, 1500, 1350, 1150, and 1000°F.

Figure 6

M 1469-1

Typical fracture which occurred in some Zr-U specimens within 24 hours after being upset to an 88 per cent reduction in thickness at various temperatures. From left to right: 50 and 70 w/o Zr-U specimens upset at 1600°F.

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Figure 7 Hardness of hot malleability specimens as a function of amount of upset at various temperatures.