An Investigation Of
SCALING OF ZIRCONIUM AT ELEVATED TEMPERATURES

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SCALING OF ZIRCONIUM

Current Work

During the last quarter, instantaneous scaling rates of zirconium were calculated for various media and duplex treatments in order to obtain a better understanding of the scaling mechanism when the dimensions of the piece are changing during the course of scaling. This work is still in progress and will be incorporated in the next technical report.

➤ Work is now underway to determine the distribution of gas (oxygen and nitrogen) reacting with zirconium. During scaling, the gas is distributed between the scale and the metal. The gas absorbed in the metal is confined to the outer layers, penetrating to a depth which depends upon the scaling temperature, time and atmosphere. ◀ This is indicated by the preliminary metallographic results in Table I for scaling in air, nitrogen and oxygen.

These early results show that:

(a) The depth of gas penetration during scaling in air generally increases with time. However, in the temperature range where growth occurs (600° - 900°C), the depth of the diffusion zone apparently remains constant, once the breakaway in the scaling rate is obtained. This is shown graphically in Fig. 1 for a scaling temperature of 900°C . At high temperatures the diffusion zone increases with time at temperature until the metal is saturated with gas.

(b) The amount of gas dissolving in the metal just before the break in the scaling rate occurs at 900°C is the same, percentagewise (15%), as that dissolving in the metal after the break in the scaling

rate. These values were calculated from the gas content of the remaining metal, Table II, the volume of the remaining metal and using a density of 6.5 gms per cu cm. for zirconium. The values obtained by this method are in good agreement with those obtained by subtracting the weight of gas (essentially oxygen) combined with zirconium as ZrO_2 from the total weight gain. The weight of gas combined with zirconium may be obtained by metallographic means by consideration of the decrease in metal volume on scaling. This latter method will be employed in future studies to determine the distribution of gas to metal and to scale supplemented by chemical analyses.

(c) In the case of scaling studies in pure oxygen and pure nitrogen the depth of the diffusion zone apparently increases continuously with time at temperature until the metal is saturated, see Table I, and with oxygen penetrating much faster than nitrogen. Gaseous penetration in the metal is about the same for air and oxygen media for a scaling time less than that necessary for a breakaway in the scaling rate in air.

Future Work

Construction of instantaneous scaling rate curves and the determination of the distribution of gas to metal and to scale will continue. A technical report will be prepared covering the work completed in 1956.

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TABLE I: Depth of Gas Penetration
on Scaling of Zirconium in Air

<u>Temp, °C</u>	<u>Scaling Time, hrs.</u>	<u>Depth of Gas Penetration on Scaling, cm.</u>			<u>Critical Time for Breakaway in Scal- ing Rate in Air, hrs.</u>
		<u>In Air</u>	<u>In Oxygen</u>	<u>In Nitrogen</u>	
700	427	.005			290
	609	.006			
750	101	.0065			80
	292	.0075			
800	2	.0016			20
	6	.003			
	21	.005			
	54	.008			
	72	.007			
900	.1	.0014			1
	.2	.0027			
	.3	.0034			
	.4	.0038			
	.5	.0046			
	.6	.0044			
	.7	.0052			
	.75	.0045			
	.8	.006			
	.9	.0063			
	1.0	.0064			
	1.1	.0064			
	1.2	.0072			
	1.25	.005			
	1.3	.0068			
	1.4	.0075			
	1.5	.0075			
	1.6	.0083			
	1.75	.0079			
	2	.005	.010		
	3	.005			
	3.56	.011			
	4.57	.005			
	5	.005			
	5.33	.0046			
	5.5	.0046			
	5.57	.0045			
	9.29	.004			
	10.15		.021		
	16		.026		
	28		.028*		
	36			.0057	
	46.7	.004-.035**			

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Table I Cont'd.

<u>Temp, °C</u>	<u>Scaling Time, hrs.</u>	<u>Depth of Gas Penetration on Scaling, cm.</u>			<u>Critical Time for Breakaway in Scaling Rate in Air, hrs.</u>
		<u>In Air</u>	<u>In Oxygen</u>	<u>In Nitrogen</u>	
900	108			.012	
	118		.034*		
950	.75	.005			1 ⁺
	2.67	.008			
	4.52	.010			
1000	1.08		.011		3
	3.5		.021		
	3.95	.018			
	6.2	.020			
1100	1.45		.018		No breakaway
	2.73	.017			
	5.98	.025			
	7	.042			
	49			.026	
1200	3.07	.042			No breakaway
1300	1.19	.038*			No breakaway

*Depth of gas penetration limited by metal thickness, i.e., the metal was completely saturated with gas under these conditions.

** 1/2" diameter sphere. Where extensive cracking of the scale occurred, the depth of gas penetration was .004 cm. Gas penetration of .035 cm was observed in those areas where the scale was relatively coherent with the underlying metal.

TABLE II: Distribution of Gas on
Scaling of Zirconium in Air

Scaling Conditions	Total Weight Gain, mg.	Chemical Analysis of Metal, Wt. %		Weight of Gas, mg.	
		Oxygen	Nitrogen	Diss'd in Metal	To Scale*
900°C, air, 0.75 hrs.	34	0.130	0.0032	5(15%)	29(85%)
900°C, air, 5.5 hrs.	250	1.17	0.129	37(15%)	213(85%)

Note: Initial specimen dimensions were about 1 3/4 x 2 x 0.16 cm.

*Scale analysis (weight percent): 25.34 oxygen; 0.201 nitrogen.

FIG. 1. DEPTH OF DIFFUSION ZONE DURING SCALING OF ZIRCONIUM IN AIR AT 900°C.