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RESEARCH ESTABLISHMENT
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OXIDATION OF 2¼% Cr, 1% Mo STEEL IN CARBON DIOXIDE

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ABSTRACT

Alloy steels were studied to find types suitable for nuclear use in carbon dioxide-cooled reactors at temperatures in the range 400 – 600 °C.

The corrosion of 2¼% Cr, 1% Mo steel in carbon dioxide was measured in the temperature range 460° to 525 °C and the gas pressure range 0 to 225 p.s.i.g. The effect of gas velocity, specimen surface treatment, and water content of the gas were also determined.

Adherent oxide scales of the $Fe_3O_4 - Fe_2O_3$ type were formed under semi-static conditions together with an unidentified spinel. A Cr_2O_3 film was never formed under any conditions. Weight gain depended mainly on temperature and varied little with gas pressure, surface treatment, or water content of the gas.

The relationship between weight gain and time varied between parabolic and cubic with weight gains ranging from 1.7 mg/cm^2 to 5.7 mg/cm^2 after 1000 hours exposure within the temperature range investigated. Estimated penetration depths after 10,000 hours ranged from 6.8×10^{-4} to 18.5×10^{-4} inches.

In high velocity gas, that is, at 150 ft/sec, weight gains varied from 0.7 mg/cm^2 at 460 °C to 2.0 mg/cm^2 at 525 °C after 100 hours. No scaling occurred during any experiment not involving thermal cycling but this may have been due to the short duration of the tests. The relative thickness of the outer Fe_2O_3 layer in the scales examined was much greater under flowing gas than under semi-static conditions.

It appears that the limiting temperature for 2¼% Cr, 1% Mo steel in carbon dioxide-cooled reactors is 490 °C; above this temperature excessive scale formation occurs which, on flaking, could seriously contaminate any gas circuit.

CONTENTS

1. INTRODUCTION

The reactor concept currently under consideration by the Australian Atomic Energy Commission is a high temperature carbon dioxide – cooled system operating at gas outlet temperatures higher than 500 °C.

Since the use of mild steel in carbon dioxide is limited to 410 °C by creep and corrosion characteristics it has been necessary to investigate the oxidation resistance of alloy steels with a view to their use in the gas ducting and heat exchanger sections of the active circuit of any reactor.

Low alloy steels containing 1 – 2% chromium and ½ – 1% molybdenum are suitably creep resistant, somewhat cheaper than the stainless steels, and only slightly more difficult to weld than mild steel. Owing to their good creep resistance they have been used widely in high temperature steam raising plant.

This report deals with the oxidation resistance of a nominal 2¼% chromium 1% molybdenum steel to carbon dioxide gas in the temperature range 460 – 525 °C.

2. EXPERIMENTAL METHODS

The steel used in all tests was supplied by Babcock and Wilcox (Aust.) Pty. Ltd., and had the following composition:

Cr	2.5%	Mn	0.7%
Mo	1.0%	Si	0.6%
C	0.22%	Cu	0.1%

Carbon dioxide, supplied from a six ton storage vessel at 300 p.s.i.g., contained two main impurities; water (< 20 p.p.m.) and air (< 100 p.p.m.). The effects of the following variables were studied within the ranges listed:

Temperature:	460 – 525 °C
Pressure:	0 – 225 p.s.i.g.
Moisture content of gas:	0 – 28,000 p.p.m.
Gas velocity:	0 – 200 ft/sec.
Surface preparation:	four different types: (a) surface ground (b) surface ground and vapour blasted (c) surface ground and etched (d) surface ground and abraded

Vapour blasting was done with a high velocity water jet containing 400 grit Al_2O_3 and the abraded specimens were polished with 400 grit SiC paper. Etching was done in boiling 50% hydrochloric acid for 5 minutes.

Pressure tests were carried out in autoclaves similar to those described by Draycott and Smith (1960) except for their greater size which resulted in better temperature profiles. Temperatures were controlled to within ± 5 °C with a Cambridge controller and variable transformer. Time from energising the furnace to attaining test temperature was usually of the order of 40 minutes. Each weight–gain value quoted in the results was the mean of between six and eighteen specimens, each of which had a surface area of approximately 7 cm^2 .

Specimens were weighed after 100 hours and 250 hours exposure and subsequently every 250 hours to 1000 hours, one test being continued for 3,500 hours. A purge of 500 ml/min of gas was maintained on all autoclaves.

The velocity effect was investigated in the small pumped loop used by Draycott and Smith (1960). Rod type specimens 4 inches long and 0.3 inches in diameter, were tested in a venturi–type section where a modified Reynolds number of 2380 (based only on local velocity and throat geometry) was obtained at the lowest velocity investigated (50 ft/sec). The gas pressure of the loop was maintained at 5 p.s.i.g. to prevent any back diffusion.

Atmospheric pressure tests were carried out in horizontal resistance furnaces wound on tubular silica formers, the specimens being supported on alumina boats. A very slight gas pressure was again maintained to prevent back diffusion.

Stanton thermobalances (deci-milligramme model) were used to investigate the effect of varying the water content of the gas and also to study the effect of oxygen on the metal.

The effect of thermal cycling on the flaking characteristics of the alloy was investigated to a limited extent in a small autoclave. In each cycle the specimens were heated to operating temperature in 30 minutes under a gas pressure of 150 p.s.i.g. and then cooled to 100 °C over a 60 minute period by means of a rapid gas purge.

The oxide scales on representative specimens were examined both metallographically and by X-ray diffraction techniques.

3. RESULTS AND DISCUSSION

3.1 General

The reaction of carbon dioxide with this steel produced a three-layered scale (Figure 1) at all exposure times greater than 100 hours; however when the temperature exceeded 525 °C the triple scale was formed during the first 100 hours in the thick sections of the scale.

Only two constituents, Fe_2O_3 and Fe_3O_4, were identified in the three layers. The outer scale was identified as Fe_2O_3 by its anisotropy to polarised light and X-ray diffraction showed only Fe_3O_4 to be present in the remaining scales. However it is thought that the middle layer was Fe_3O_4 and the inner layer a mixed Fe - Cr spinel with a lattice parameter close to Fe_3O_4. Some scales also contained unoxidised metal in the inner layer.

The thickness of the Fe_2O_3 layer tended to decrease with increasing gas pressure (Figures 1 and 2) while porosity at the Fe_2O_3 - Fe_3O_4 interface was greater both at lower pressures and under the effect of flowing gas (Figures 3 and 4). The thickness of the Fe_2O_3 layer relative to the total scale was greater under the influence of velocity than under semi-static conditions. At the same temperature and pressure (490 °C and atmospheric) 14 per cent. of the total scale was Fe_2O_3 both after 500 hours at 116 ft/sec (Figure 4) and after the longer period of 1250 hours under semi-static conditions (Figure 3). Thin Fe_2O_3 layers were detected by electron diffraction.

The greatest thickness of any scale formed during the experiments was 34 microns after 3500 hours at 490 °C and 75 p.s.i.g. Generally, total scale thickness was directly proportional to weight gain/cm^2 and in no case was any Cr_2O_3 formed. No flaking was experienced during any of the experiments with carbon dioxide as the reactant gas, except under thermal cycling conditions.

In some experiments, oxygen and air were used in place of carbon dioxide. In most of these experiments the Stanton thermobalance was used in an endeavour to determine whether the back diffusion of air had any noticeable effect on weight gain. All tests were at atmospheric pressure.

Draycott and Smith (1960) showed theoretically that there should be no marked difference in the oxidation rate of an alloy steel in carbon dioxide or air provided that no difference in nucleation characteristics exists. However, we found that with both oxygen and air the rate of attack as measured by weight gain was much slower. However severe flaking occurred after 500 hours and the experiments were discontinued.

Morozova and Ipat'ev (1956) showed that at 850 °C to 1000 °C both water vapour and carbon dioxide oxidise a 12% Cr steel faster than does oxygen or air, oxygen being three to four times faster than air. Our tests showed no noticeable difference between air and oxygen at 575 °C.

In general it was found that scales formed by carbon dioxide were much thicker than those formed by oxygen (Figures 5 and 7). Oxygen scales showed evidence of a fourth and unidentified layer in the outermost part (Figure 7) and they were devoid of fine porosity in the innermost part, unlike the specimens exposed to carbon dioxide at atmospheric pressure. The formation of Fe_2O_3 was less in oxygen than in carbon dioxide and only when the temperature was above 525 °C did it form during the first 100 hours; otherwise it formed at between 100 and 250 hours.

3.2 Effect of Surface Preparation

The first variable investigated was the effect of surface preparation since the possible elimination of some surfaces would greatly reduce the number of experiments needed for full study of the steel. Two 1000 – hour tests were then completed at 525 °C and pressures of 75 and 150 p.s.i.g.

In each test four batches of eight specimens were examined, the batches representing the four different surface treatments. It can be seen from Figure 8 that the effect of surface treatment on weight gain was negligible, with a maximum variation of 9 per cent., while specimen to specimen variation within a batch was often 10 – 20 per cent. The only result of interest was an initial difference in the effect of different surfaces but as time increased it became negligible. Because the effect was small the results which follow were obtained with surface ground specimens.

3.3 Effect of Temperature and Pressure

For the three temperatures and four pressures investigated, the weight gain–time relationship as shown in Figure 9 varied between parabolic and cubic. It was clearly evident that while weight gain increased with temperature no clear relationship between weight gain and pressure could be deduced. However, it did appear that the greatest weight gain was evident at atmospheric pressure and could not be wholly caused by back diffusion of air as described in Section 3.5 below.

An interesting fact arose when comparing 2¼% Cr, 1% Mo steel and 1% Cr, ½% Mo steel. Draycott, Fox, and Hubery (1962) showed that the higher chromium steel at 525 °C and 150 p.s.i.g. gave 11 per cent. greater weight gain after 1000 hours exposure even though it contained twice the amount of chromium, whereas at 460 °C both alloys had similar weight gains. In a test of extended duration (3500 hours) at 490 °C and 75 p.s.i.g. (Figure 10) a linear relationship, such as was observed in long term tests on the 1%Cr, 0.5% Mo steel did not exist at any time up to 3500 hours.

The relationship between weight gain and time of exposure was found to be best represented by the equation:

$$\Delta W^n = kt ,$$

where ΔW = weight gain in mg/cm^2
t = exposure time in hours
and k = rate constant.

All weight gain data were plotted logarithmically against time, each straight line being fitted to the experimental points by the method of least squares. The results are shown in Figures 11, 12, 13, and 14. Values for n and k obtained from these graphs are listed in Table 1. The value of n ranged from 2.24 to 2.88 while k values were between 0.004 and 0.132 $(mg/cm^2)^n/h$ respectively. These values agree closely with those obtained for the lower alloy steel (Draycott and Hubery 1961).

The rate constant k and the reciprocal absolute temperature were then fitted to the Arrhenius equation

$$k = Ae^{-\frac{E}{RT}} ,$$

again by the method of least squares, E being activation energy and A the frequency factor. These results are shown in Figure 15. In the extended duration test of 3500 hours at 490 °C and 75 p.s.i.g., straight lines were obtained when time was plotted against both the second and third powers of weight gain, indicating a relationship varying between parabolic and cubic over this extended time.

From Table 1 it is apparent that the relationship between weight gain and time varied between cubic and parabolic with no particular trend to either relationship when pressure or temperature were varied within the ranges studied. From thermobalance studies it was found that a strict parabolic relationship existed up to approximately 50 – 100 hours and thereafter a cubic relationship best fitted the data. Generally a decrease in the reaction rate occurred at approximately 24 hours and again at about 50 hours, after which the rate remained reasonably constant. These rate changes are shown in Figure 16.

Metallographic examination revealed that this steel was very similar to 1% Cr, ½% Mo steel as reported by Draycott and Hubery (1961). With increasing temperature, heavier scales were formed but the scale always consisted of the same three constituents.

The effect of gas pressure was only very slight, thicker scales being formed at higher pressures. After short exposures, only the two variations of the Fe_3O_4 spinels were formed but on longer exposure Fe_2O_3 was formed as the outer layer. Photomicrographs of representative scales are shown in Figures 1, 2, and 6. In all experiments the initial scale formed was light chocolate in colour and had a "suede-like" texture. Unlike mild steels, as reported by Huddle and Wyatt (1957), this colour and texture began to disappear after 250 hours when the colour changed to a light matt grey; after 1000 hours the specimens usually developed a dark grey to black matt finish.

The depth of penetration of the oxide film below the original metal surface was calculated by extrapolating the weight gain to 10,000 hours and by assuming the oxides formed to be of the type M_3O_4 with M having an atomic weight of 56. Metal density was taken as 7.8 g/cm^3.

Penetration was found to increase with increasing temperature but was completely unaffected by any change in pressure between atmospheric pressure and 225 p.s.i.g. These results are given in Table 2.

3.4 Effect of Velocity

In studying the effect of velocity three different temperatures were investigated; 460, 490, and 525 °C as well as four different velocities; 50, 100, 150, and 200 ft/sec. Most experiments lasted 100 hours. Nominal Reynolds numbers for the four velocities at 525 °C were 2380, 4760, 7140, and 9520 respectively so that all but the lowest velocity were within the turbulent range.

The weight gain – gas velocity relationship is shown in Figure 17; the weight gain values for zero gas velocity being those of the previous atmospheric tests. The hatched bands show the maximum variation in weight gain (after 100 hours exposure) of all the atmospheric pressure tests. Each point represents only one velocity specimen the surface area of which is approximately 22 cm^2.

For 460 °C the straight line was fitted to the data by the method of least squares. It shows that increasing velocity results in decreasing weight gain. However for 490 °C and 525 °C the points are so scattered that no conclusion can be reached as to the relationship between velocity and weight gain. These results differ from those obtained by Draycott and Hubery (1961) for the 1% Cr, ½% Mo steel where scaling was induced at high velocities in a systematic fashion.

Owing to a very large scatter in the results, especially as temperature increased, the scale thickness on each of the 490 °C specimen rods was measured, as it was suspected that transient effects such as a moving vena-contracta may have been causing localised corrosion.

Figure 18 shows the relationship between film thickness and distance along the length of the specimen. The peaks and hollows on the graphs indicate that some flow disturbance did occur at varying positions depending on the gas velocity.

No flaking occurred during any of the experiments and metallographic examination as shown in Figure 4 revealed scales similar to those formed on the semi-static specimens. At 116 ft/sec and 490 °C a 500 hour exposure resulted in a scale thickness of 21 microns (3 microns being Fe_2O_3) and a weight gain of 3.28 mg/cm^2, compared with 2.92 mg/cm^2 on a similar semi-static test at atmospheric pressure. The outstanding feature of the scales formed under dynamic conditions was that the Fe_2O_3 layer was considerably thicker (relative to total scale thickness) than in the semi-static tests. Much greater porosity was evident at the $Fe_2O_3 - Fe_3O_4$ interface under dynamic conditions, so any scaling which might have occurred would probably only result in the loss of Fe_2O_3 and not the bulky Fe_3O_4 scale.

3.5 Effect of Gas Impurities

The main gas impurity studied was water vapour. This was done on Stanton thermobalances over 100 hour periods. For 28,000 p.p.m. moisture levels, the gas was passed through water bubblers and for $<$ 5 p.p.m. it was dried through beds of molecular sieves. All experiments were done at atmospheric pressure.

The weight gain in wet gas at 575 °C was 14 per cent. greater than that in the dry gas as shown in Figure 19 but at 550 °C this difference was only 2.6 per cent. At 525 °C the effect was reversed and the dry gas caused a weight gain 3.3 per cent. greater than that in the wet gas.

Some experiments were also done using oxygen, the other main impurity in the carbon dioxide used. The results were quite remarkable; at 550 °C in dry oxygen the weight gain was only 0.9 mg/cm^2 or 64 per cent. smaller than that in dry CO_2. At 525 °C dry oxygen caused a 15 per cent. greater weight increase than wet oxygen and again the weight gain was 68 per cent. smaller than that in dry carbon dioxide. When these results for oxygen were checked over longer periods it was found that at about 500 hours at 525 °C severe flaking occurred.

Metallographically, the scales formed in oxygen were much thinner than those in carbon dioxide (Figures 5 and 7) however the former showed some evidence of a fourth and unidentified layer in the outermost layer. Fe_2O_3 formation was less in oxygen than in carbon dioxide; above 525 °C it formed in the first 100 hours; below this temperature it formed at between 100 and 250 hours. Scales formed in oxygen did not show any of the fine porosity in the innermost layer which was characteristic of the scales formed in carbon dioxide at atmospheric pressure.

All these tests showed that only above 550 °C did water vapour have any detrimental effect. The results also proved that if any back diffusion of air did occur in any of the atmospheric pressure thermobalance tests its effect would have been inhibitive. However since tests at atmospheric pressure, in properly sealed equipment gave the same results as the thermobalance tests, it was conclusively proved that no back diffusion occurred.

3.6 Thermal Cycling

Two series of thermal cycling tests were run, one of 400 cycles (100 °C to 525 °C), the other of 500 cycles (100 °C to 490 °C). In each case the gas pressure was 150 p.s.i.g. and six specimens were exposed, one of which was removed for examination after every 100 cycles.

At 490 °C slight flaking occurred after 500 cycles, but at 525 °C flaking started at 300 cycles and became more pronounced after 400 cycles. Photographs of sample specimens are shown in Figure 20.

Two specimens which had previously been exposed for 1000 hours to carbon dioxide at 460 °C and 75 p.s.i.g. with a resultant weight gain of 2.5 mg/cm^2 were subjected to 100 cycles at 525 °C and 150 p.s.i.g. but no flaking occurred.

Thus if the limiting temperature for this steel is 490 °C, flaking could begin after 500 cycles.

3.7 Type of Reaction

The reaction between 2¼% Cr, 1% Mo steel and carbon dioxide over a 1000 - hour period gave rise to four distinct reaction rates involving two distinct types of reaction.

At 525 °C and atmospheric pressure the relationship between weight gain and time was a simple parabolic type over the first 100 hours (Figure 16); thereafter the data were best represented by a mixed parabolic equation. The reaction rates and reaction types for this steel over the first 1000 hours of exposure are listed below:

Time hours	Reaction Rate (k) $(mg/cm^2)^n/h$	n	Reaction Type
4 - 24	0.040	2	$(\Delta W)^2 = kt$
24 - 50	0.034	2	$(\Delta W)^2 = kt$
50 - 100	0.317	2	$(\Delta W)^2 = kt$
100 -1000	0.151	3	$(\Delta W)^3 = kt$

During the 100 - 1000 hour period the experimental results were best expressed by the equation $(\Delta W)^{2.5} = kt$. However the equation $(\Delta W)^3 = kt$ represented the data more accurately than did $(\Delta W)^2 = kt$ indicating that the relationship was tending to be cubic rather than parabolic.

Metallographic examination of specimens exposed for periods up to 100 hours revealed the formation of a duplex-layered scale in which one layer was growing outwards and the other inwards, the interface between the layers being the original metal surface. X-ray diffraction showed only Fe_3O_4 to be present and it is thought that the outer layer may have been Fe_3O_4 and the inner layer a mixed Fe-Cr spinel, with a lattice parameter very close to Fe_3O_4. At a later stage Fe_2O_3 formed as a third layer on the outside of the scale and was readily detectable by X-ray diffraction techniques.

The growth of the scale proceeds by inward diffusion of oxygen ions and outward diffusion of iron ions through these layers. Further work is required to establish the exact mechanism of the scale formation processes.

Evans (1960) states that while the oxidation rate is controlled purely by movement of ions through the oxide film a simple parabolic relationship exists between film thickness and time. However once any boundary reaction occurs, to take partial control of the oxidation rate, the existing relationship is modified to some type of mixed parabolic law.

In our work the change in relationship from parabolic to cubic after 100 hours seems to indicate that control of the reaction is initially by diffusion of metal and gaseous ions through the oxide films but that subsequently partial control is established by some boundary reaction in conjunction with the previous diffusion control.

3.8 Possible Applications and Comparisons with other Steels

In Australia, at present, only limited use is being made of this low alloy steel. The main steam line at Yallourn Power House, fabricated from this alloy, is 14 inches o.d., 2¼ inch wall thickness, and operates at 1500 p.s.i.g. and 569 °C; the temperature is limited to avoid austenite formation.

The trend in boiler work has been towards a 1% Cr, ¼% Mo, ¼%V steel, which is more ductile and slightly superior in creep resistance to the 1% Cr, ½% Mo steel examined by Draycott and Hubery (1961). In the Vales Point Power Station the former steel is being used in large quantities for such equipment as superheater and steam outlet pipes (2400 p.s.i.g., 569 °C), hot reheat pipes (650 p.s.i.g., 569 °C), and feed water pipes (3600 p.s.i.g., 252 °C). The hot reheat pipe is 20 inch nominal bore while the steam outlet pipe is 10 inch nominal bore, both having a 2¾ inch wall thickness.

Considering mild steel (£A 60/ton) as the basis of costing, 2¼% Cr, 1% Mo steel costs approximately £A 200/ton unfabricated. Heat exchangers envisaged by Lawther and Draycott (1961) as suitable for a 30 MW(e) H.T.G.C. reactor were costed for the use of both low alloy and stainless steel materials. It was found that even disregarding the better compatibility of stainless steel, the savings by using low alloy steels appeared to be negligible.

If the superheater were fabricated from 18.8.Ti steel and the boiler and economiser from 2¼% Cr, 1% Mo steel, the cost would be £108,000. If the whole exchanger were fabricated from 2¼% Cr, 1% Mo steel, the cost could be £102,000 while the cost would be £101,000 if 1% Cr, ½% Mo steel were used.

Watkins (1959) reported a weight gain of 1.98 mg/cm^2 after 1008 hours, when mild steel was exposed to carbon dioxide at 500 °C and 118 p.s.i.g. This is approximately 40 per cent. smaller than our results on 2¼% Cr, 1% Mo steel. By comparison a G.N.E.C. report (1960) showed that carbon steel (0.28%C) SA212 showed a weight gain of 1.4 mg/cm^2 after 1000 hours in carbon dioxide at 426 °C and 150 p.s.i.g. If Watkins' data are interpolated to this latter temperature the corresponding weight gain is 0.9 mg/cm^2. Thus it appears that mild steels do show better compatibility to static carbon dioxide than 2¼% Cr 1% Mo steels but they do not have the necessary creep characteristics of the latter and hence cannot be used at such high temperatures.

Leclercq et al. (1960) tested a similar alloy which included 0.5% vanadium. This material, on exposure to carbon dioxide, showed a weight gain of 3.0 mg/cm^2 after 1000 hours at 500 °C and 375 p.s.i.g., which is about 16 per cent. lower than our figure. G.N.E.C. (1960) confirmed that in flowing carbon dioxide carbon steel corroded less than low alloy steels at temperatures up to 580 °C.

It is therefore clear that the corrosion resistance of 2¼% Cr, 1% Mo steel appears to be worse than that of carbon steel under both static and dynamic conditions within the temperature range 450 °C to 525 °C

4. CONCLUSIONS

(1) The use of 2¼%Cr, 1% Mo steel in carbon dioxide – cooled reactors at above 490 °C is not recommended. The corrosion rate is almost completely controlled by temperature, there being little effect from gas pressure and surface treatment.

(2) Adherent oxide scales are formed under semi–static conditions, but metallographic examination suggests that there is an increased possibility of scaling under flowing gas conditions. Owing to the limited duration of the velocity tests no scaling was actually observed but the Fe_3O_4 – Fe_2O_3 interface was very porous indicating that flaking at this point would be possible, with subsequent loss of Fe_2O_3. The proportion of Fe_2O_3 in the layer was always greater under dynamic conditions. We believe that longer exposure times at high gas velocity would markedly increase the reaction rate.

(3) Reaction rates at between 460 °C and 525 °C do not increase significantly due to water vapour in the gas.

(4) The oxidation mechanism is very similar to that of the 1% Cr, ½% Mo steel, and weight gains are greater than those reported for mild steel. In our experiments the scales always consisted of three layers; two types of Fe_3O_4 spinels and an outer Fe_2O_3 layer. There was no Cr_2O_3 layer in any scale examined.

5. ACKNOWLEDGMENTS

All metallographic and X–ray analyses were performed by Mr. K. Watson and Mr. J. McCracken to whom the authors wish to express their gratitude.

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TABLE 1

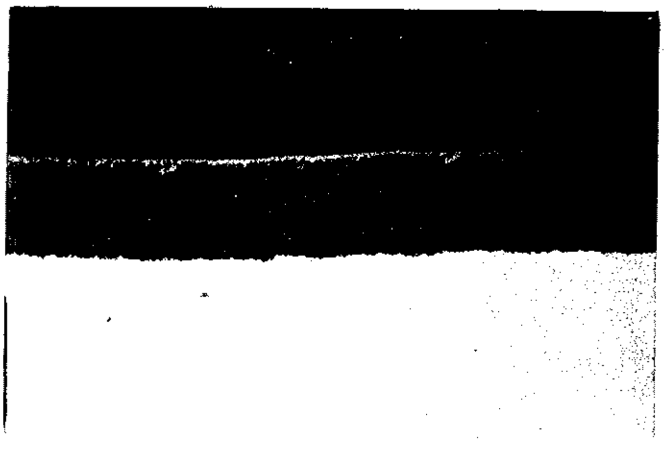
REACTION KINETIC DATA FOR 2¼% Cr, 1% Mo STEEL IN CO_2

Temp. °C	Pressure p.s.i.g.	Slope n	Rate Constant k $(mg/cm^2)^n/h$	Activation Energy E cal/g mole metal	Frequency Factor A $(mg/cm^2)^n/h$
460	0	2.882	.0132	27,830	3.16×10^6
490		2.475	.0305		
525		2.825	.1324		
460	75	2.404	.0086	18,333	4.64×10^3
490		2.597	.0276		
525		2.288	.0400		
460	150	2.237	.0038	33,667	1.0×10^8
490		2.445	.0223		
525		2.481	.0634		
460	225	2.639	.0077	27,500	2.15×10^6
490		2.564	.0223		
525		2.632	.0767		

TABLE 2

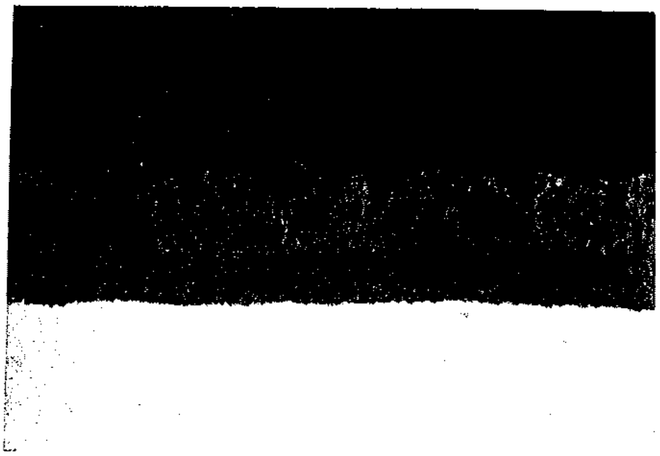
ESTIMATED PENETRATION DEPTHS BASED ON 1000 HOUR TESTS

Temp. °C	Pressure p.s.i.g.	Wt. gain after 10,000 hours mg/cm^2	Penetration depth after 10,000 hours	
			$mm \times 10^{-3}$	$in \times 10^{-4}$
460	0	5.45	18.4	7.3
490		10.07	33.8	13.3
525		13.03	43.8	17.2
460	75	6.37	21.5	8.5
490		8.71	29.4	11.6
525		13.71	46.2	18.2
460	150	5.09	17.0	6.7
490		9.12	30.7	12.1
525		13.46	45.4	17.9
460	225	5.20	17.6	6.9
490		8.22	27.6	10.9
525		12.45	42.0	16.5



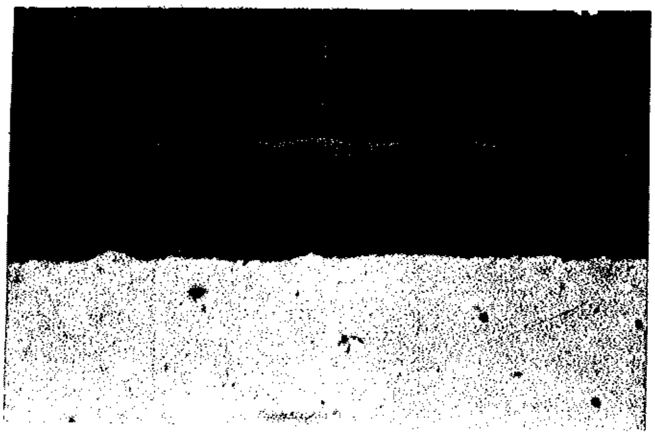
Unetched X500

FIGURE 1 1000 hours, 525 °C, 75 p.s.i.g.



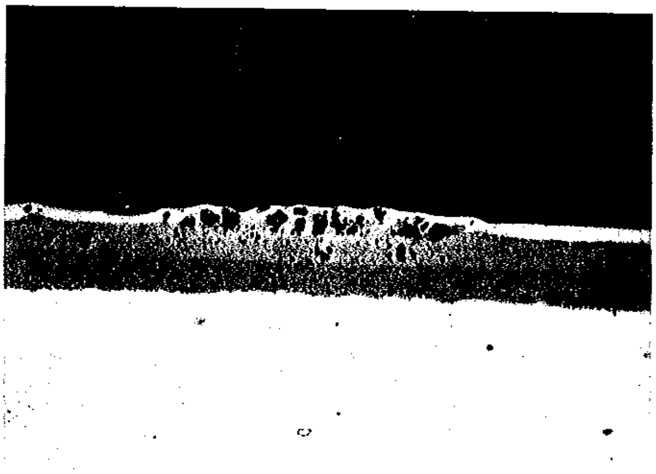
Unetched X500

FIGURE 2 750 hours, 525 °C, 225 p.s.i.g.



Unetched X500

FIGURE 3 1250 hours, 490 °C, atmospheric pressure



Unetched X500

FIGURE 4 500 hours, 490 °C, 5 p.s.i.g. 116 ft/sec

Unetched X1000

FIGURE 5 100 hours, 525 °C, atmospheric pressure

Unetched X1000

FIGURE 6 500 hours, 525 °C 75 p.s.i.g.

FIGURES 1 – 6 OXIDE SCALES ON SURFACE GROUND SPECIMENS EXPOSED TO CO_2

Unetched X1000

FIGURE 7 OXIDE SCALE ON SURFACE GROUND SPECIMEN EXPOSED TO OXYGEN FOR 100 HOURS AT 525 °C AND ATMOSPHERIC PRESSURE

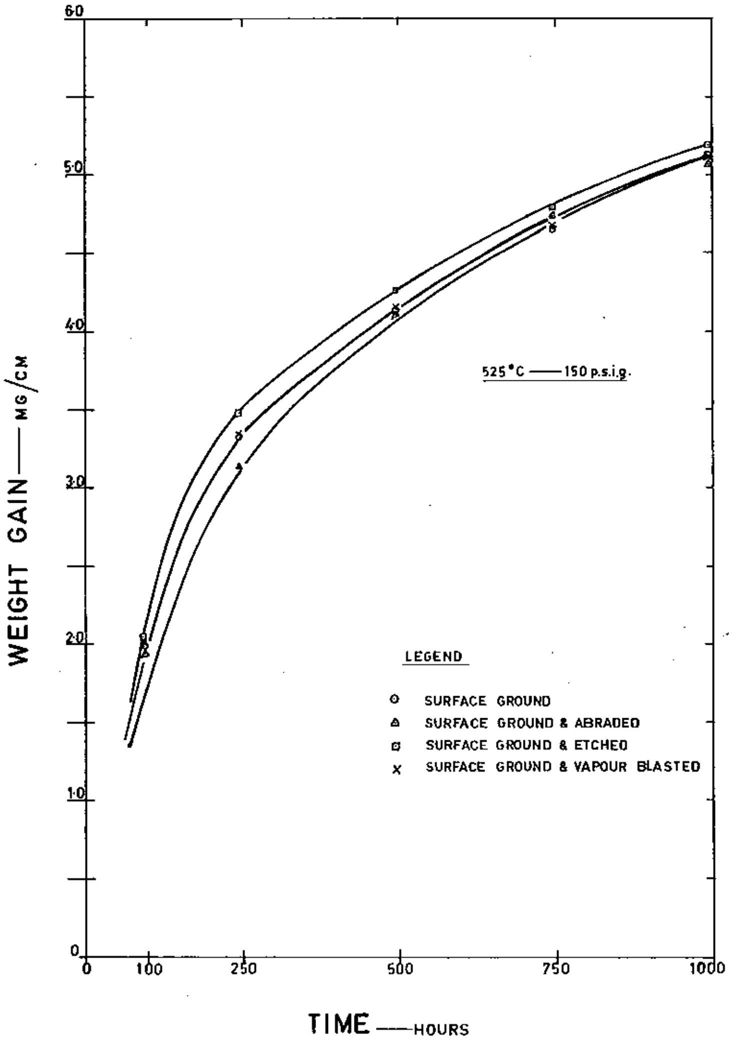


FIGURE 8. EFFECT OF SURFACE FINISH ON WEIGHT GAIN.

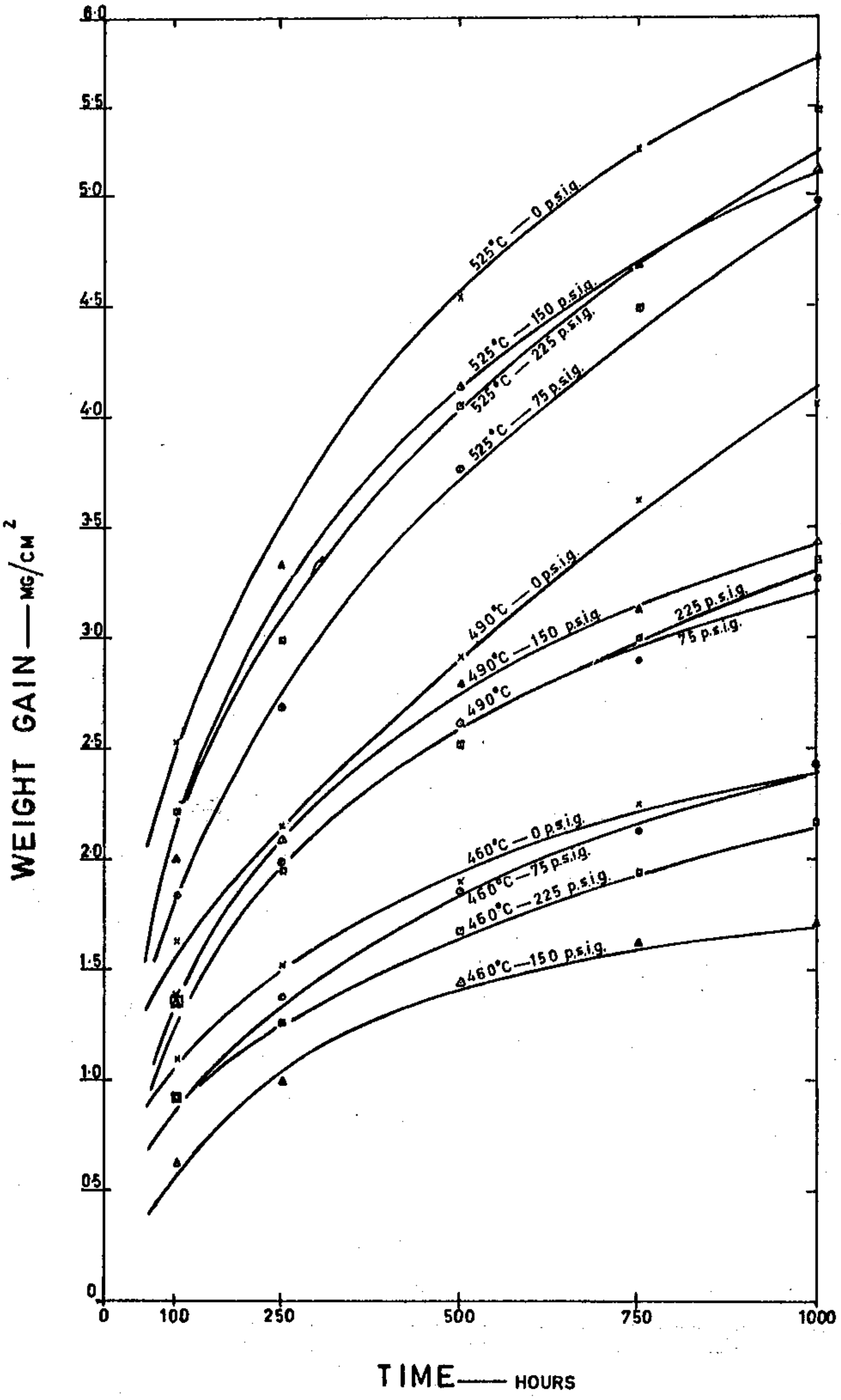


FIGURE 9. WEIGHT GAIN DATA FOR SURFACE GROUND SPECIMENS AT ALL TEMPERATURES AND PRESSURES.

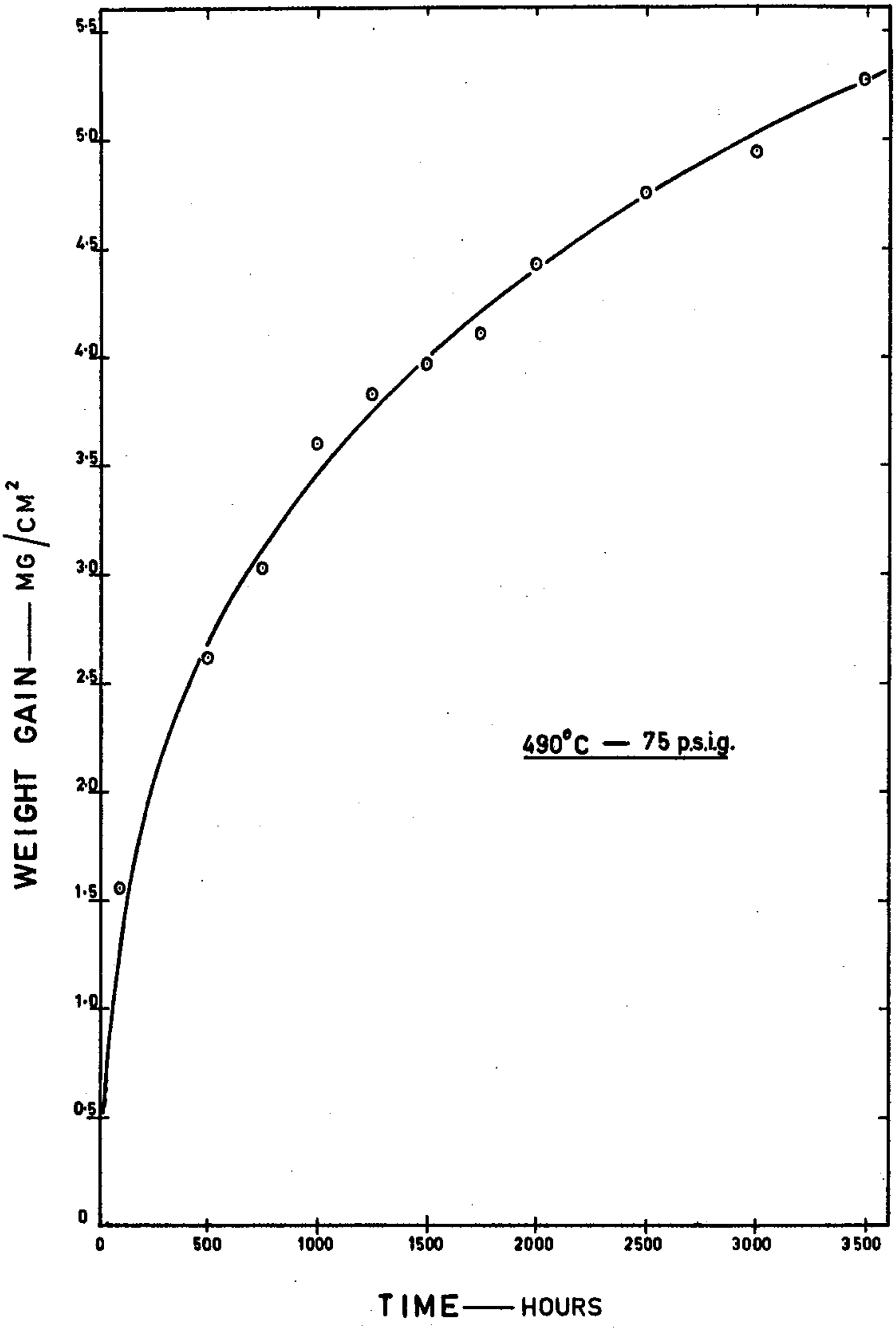


FIGURE 10. EXTENDED DURATION EXPOSURE OF 2¼% Cr., 1% Mo. STEEL IN CO_2.

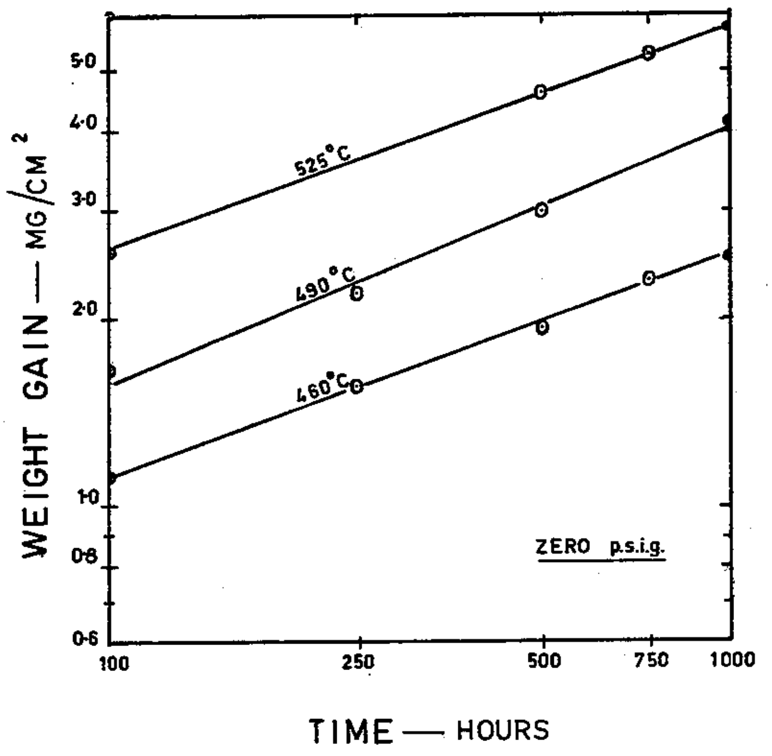


FIGURE 11. LOGARITHMIC GRAPH OF WEIGHT GAIN DATA AT ATMOSPHERIC PRESSURE

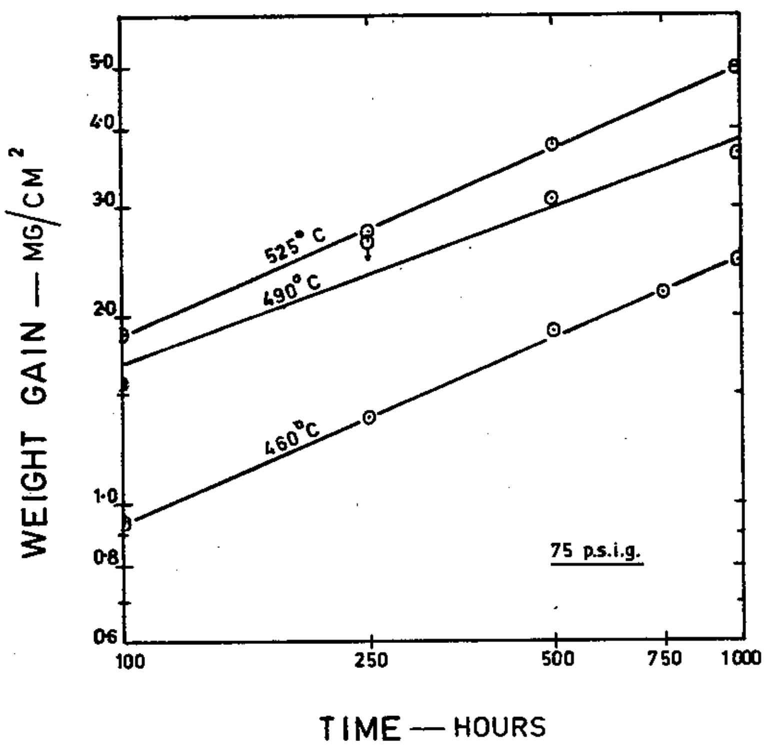


FIGURE 12 LOGARITHMIC GRAPH OF WEIGHT GAIN DATA AT 75 P.S.I.G.

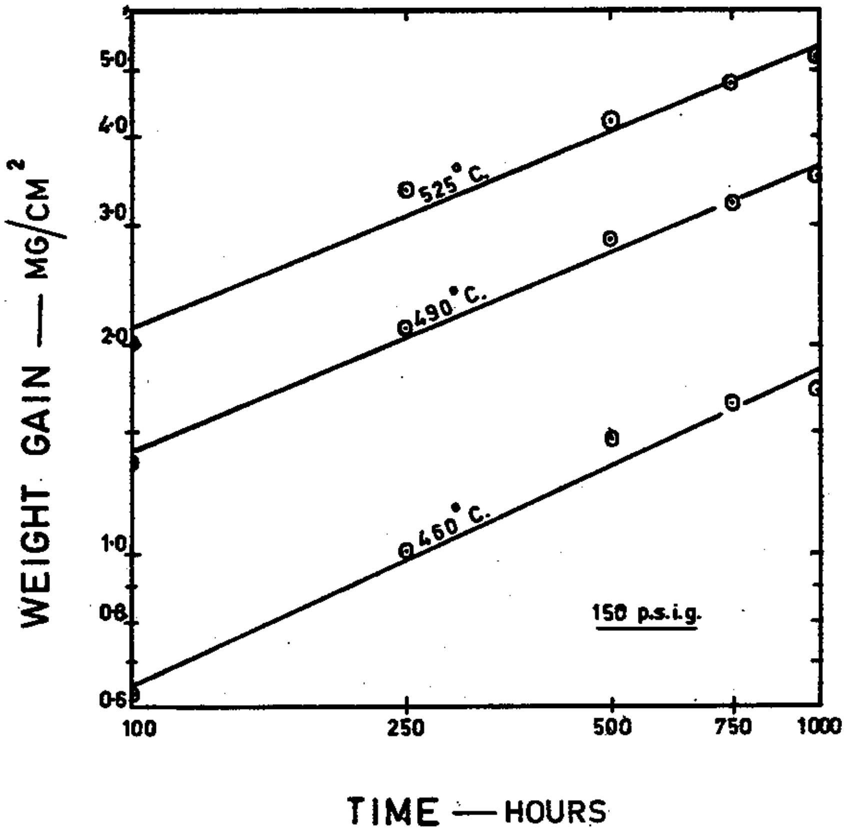


FIGURE 13. LOGARITHMIC GRAPH OF WEIGHT GAIN DATA AT 150 P.S.I.G.

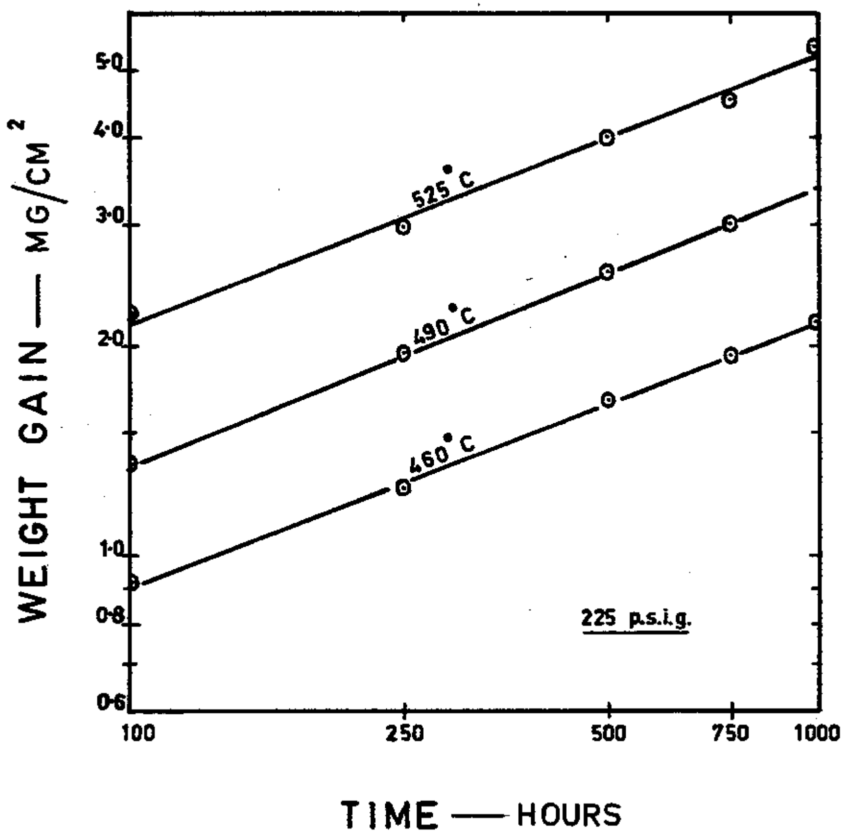


FIGURE 14. LOGARITHMIC GRAPH OF WEIGHT GAIN DATA AT 225 P.S.I.G.

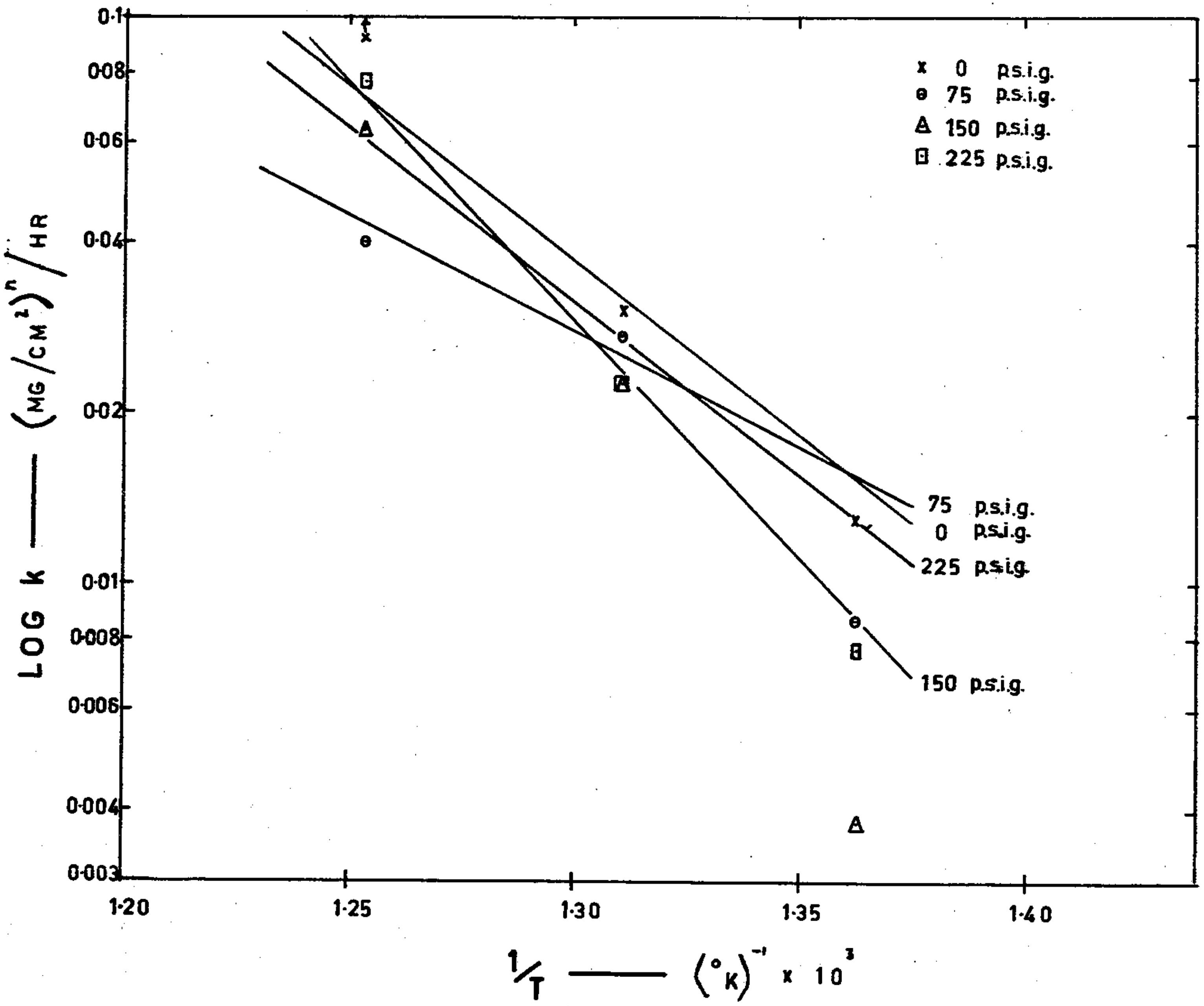


FIGURE 15. ARRHENIUS PLOT OF REACTION RATES.

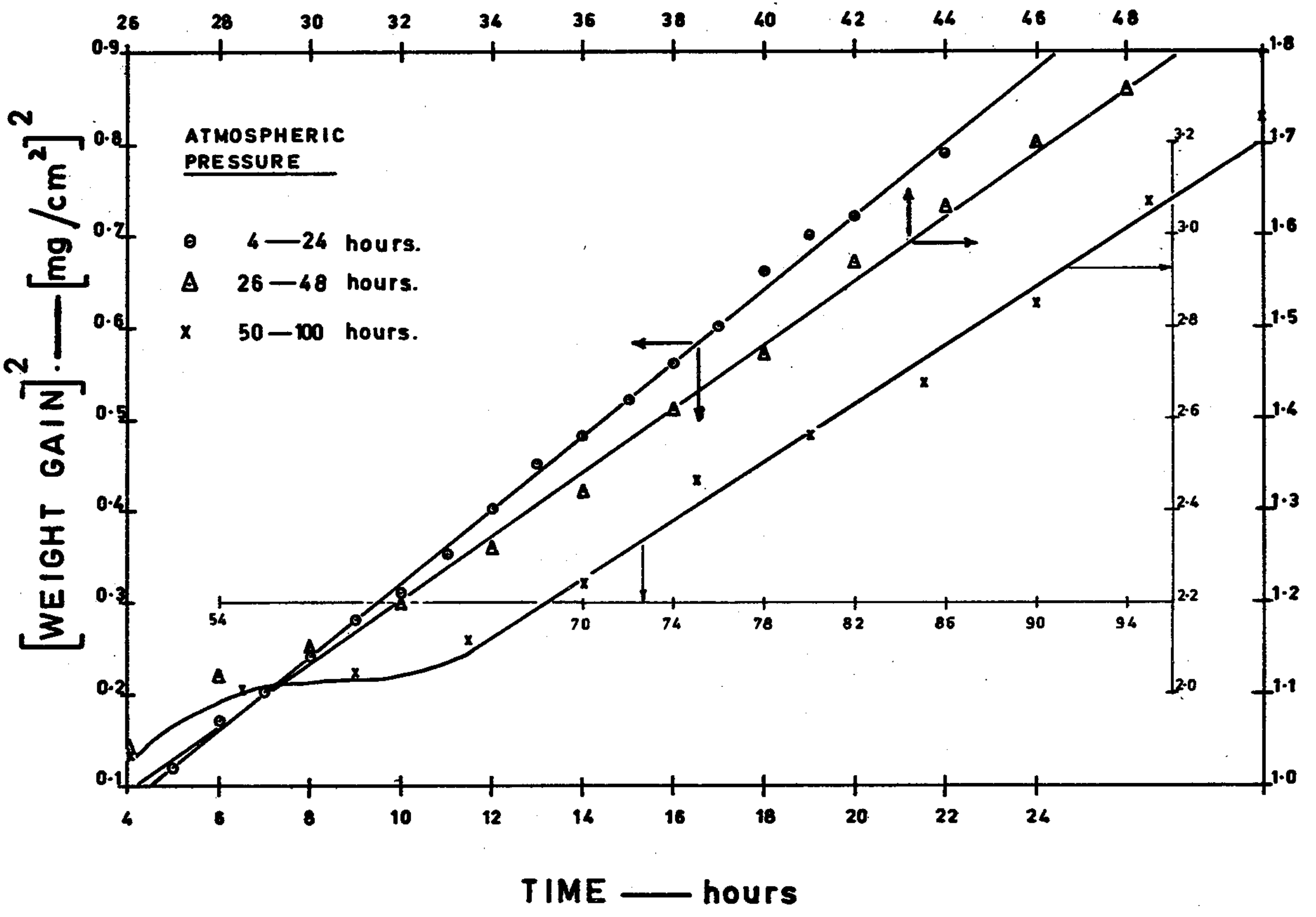


FIG. 16. RELATIONSHIP BETWEEN (WEIGHT GAIN)^2 AND TIME (0—100 HOURS) AT 525° C.

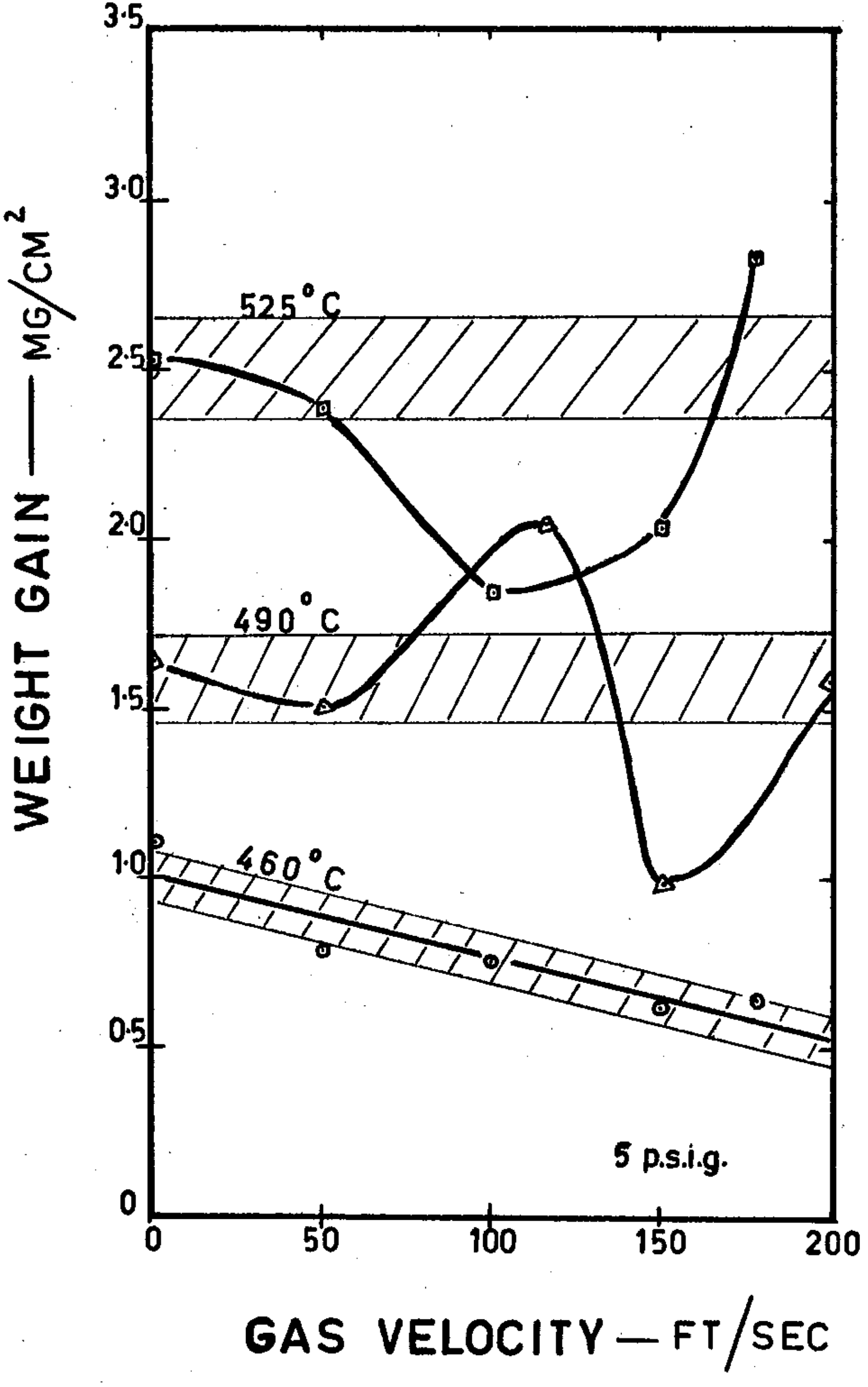


FIG. 17 EFFECT OF GAS VELOCITY ON WEIGHT GAIN AFTER 100 HOURS.

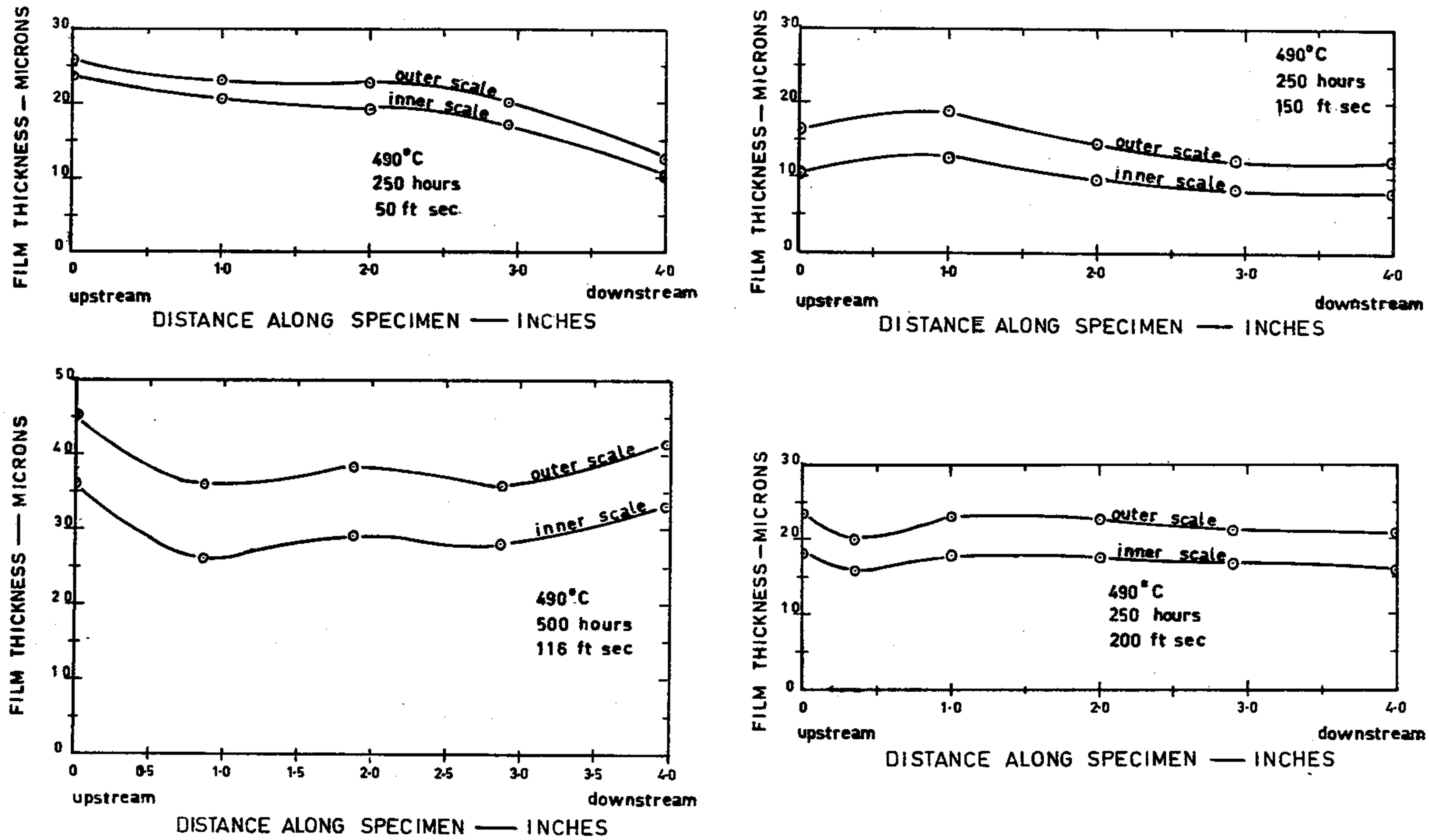


FIGURE 18. VARIATION OF FILM THICKNESS ALONG THE SPECIMEN DUE TO VELOCITY EFFECTS.

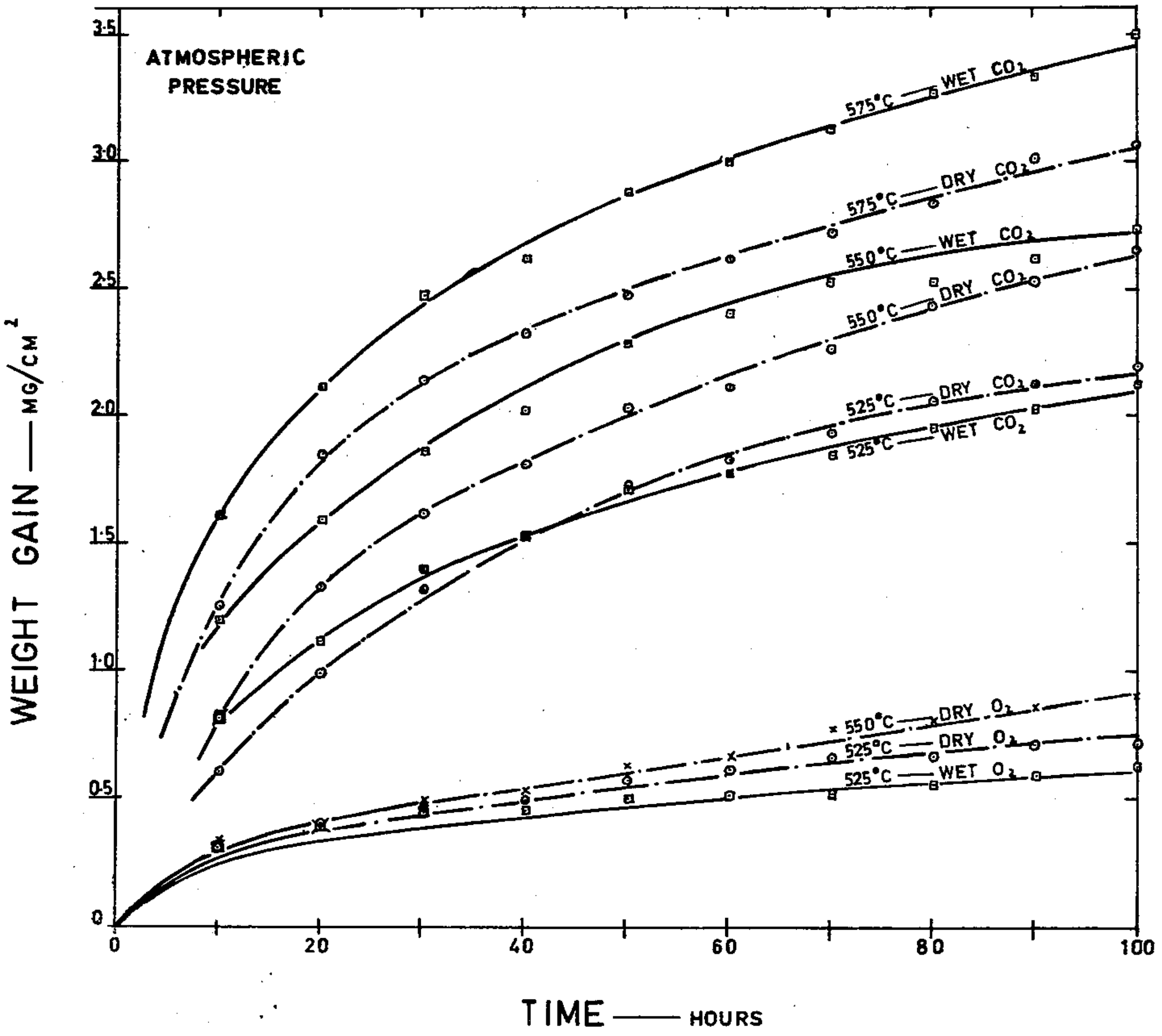
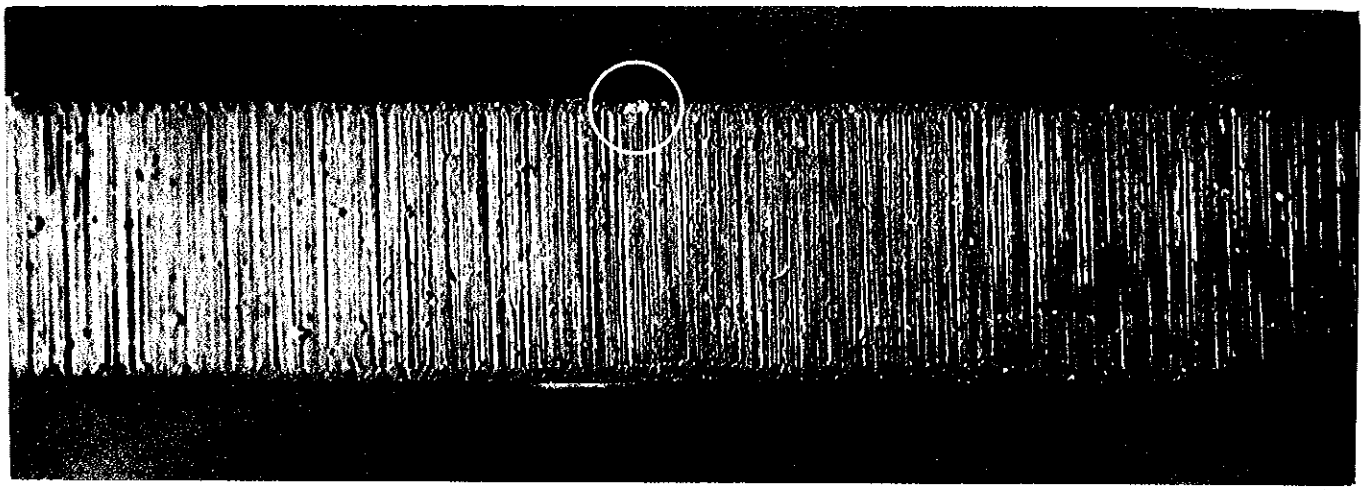
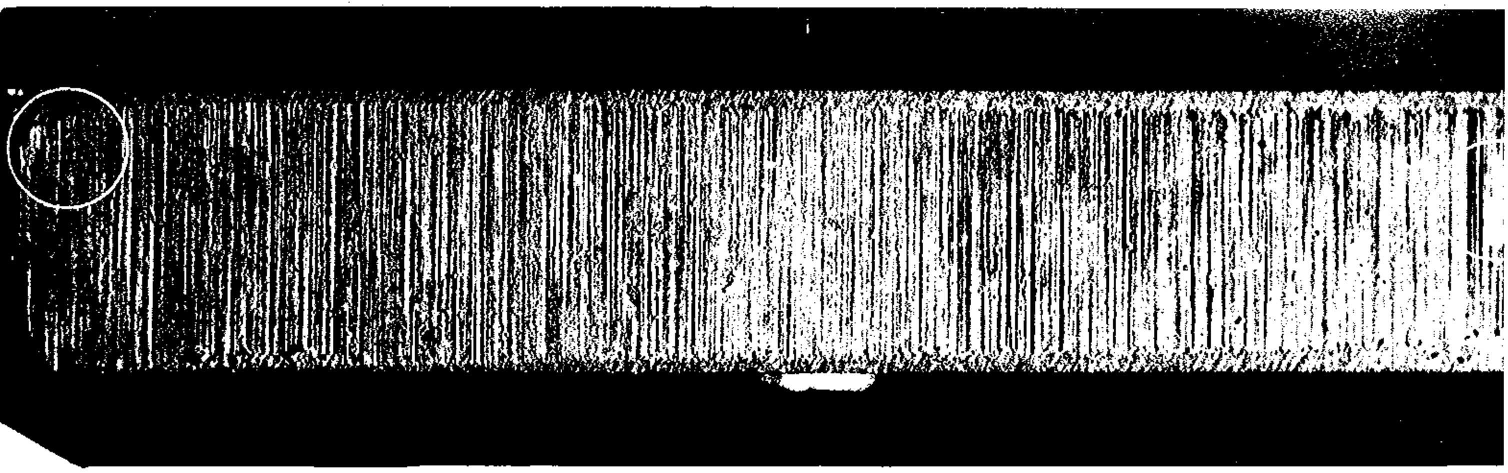


FIGURE 19. WEIGHT GAIN DATA IN DIFFERENT ATMOSPHERES



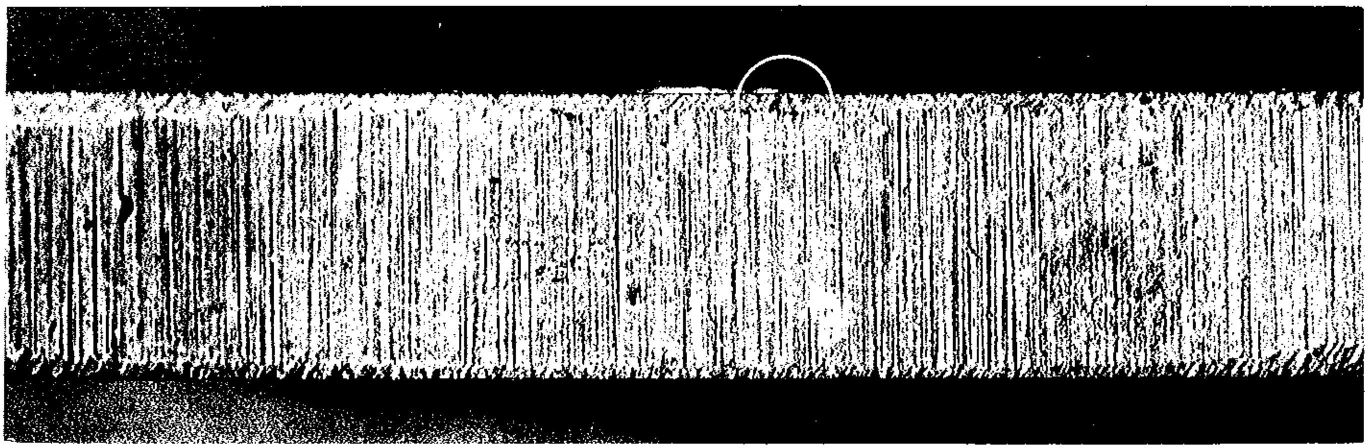
X 10

A. 300 CYCLES, 100 °C – 525 °C



X 1

B. 400 CYCLES, 100 °C – 525 °C



X 10

C. 500 CYCLES, 100 °C – 490 °C

FIGURE 20 OXIDE SCALE WHICH HAS FLAKED FROM SURFACE GROUND SPECIMEN AFTER THERMAL CYCLING IN CO_2 AT 150 p.s.i.g.