

TABLE I Non-stoichiometry in $\text{Cu}_2\text{O}_{1+\gamma}$

P_{O_2} (atm)	$\gamma \times 10^4$		[5]		[6]		This work	
	[4]		900° C	1000° C	900° C	1000° C	900° C	1000° C
	900° C	1000° C	900° C	1000° C	900° C	1000° C	900° C	1000° C
4.3×10^{-2}	—	9.1	—	11.4	7.3	13.1	—	7.4
8.5×10^{-3}	2.8	5.9	5.4	8.3	4.6	8.7	2.3	4.9
9.6×10^{-4}	1.6	3.2	3.0	5.2	2.8	5.1	1.3	2.9
1×10^{-5}	—	—	—	—	-8.77	-55	0.43	0.92

change of the sample plotted versus a $P_{\text{O}_2}^{1/4}$ dependence at different temperatures and at pressures ranging from 5×10^{-2} to 5×10^{-6} atm. From Fig. 2 it may be seen that the linear relationship extends below 10^{-3} atm pressure, at which for a temperature around 1000°C a change of the type of non-stoichiometry should occur [6]. This would indicate that the copper vacancy defect model is valid over the whole range of oxygen partial pressure considered.

The plot of $\log K$ versus $1/T$ represented in Fig. 4 gives values of A and E of Equation 3: $A = 11.9$ and $E = 22.5 \text{ kcal mol}^{-1}$. Using these values the deviation from stoichiometry in $\text{Cu}_2\text{O}_{1+\gamma}$ has been calculated for different partial pressures of oxygen and compared to values derived from results and equations in [4–6] in Table I.

In conclusion it seems that the predominant ionic defect in Cu_2O at high temperature is V_{Cu}^\times down to oxygen pressures of at least 5×10^{-6} atm, the enthalpy of formation of copper vacancies with respect to oxygen gas at 1 atm being $22.4 \text{ kcal mol}^{-1}$.

References

1. R. S. TOTH, R. KILKSON, D. TRIVICH, *Phys. Rev.* **122** (1961) 482.
2. P. KOFSTAD, "Non-Stoichiometry diffusion and electrical conductivity in binary metal oxides" (Interscience, New York, 1972) p. 328

3. H. DUNNWALD and C. WAGNER, *Z. Physik. Chem.* **B22** (1933) 212.
4. M. O'KEFFE and W. J. MOORE, *J. Chem. Phys.* **36** (1962) 3009.
5. C. WAGNER, H. HAMMEN, *Z. Physik, Chem.* **B40** (1938) 197.
6. YU. D. TRETAKOV, V. F. KOMAROV, N. A. PROSVIRNINA, I. B. KUTSENOK, *J. Solid State Chem.* **5** (1972) 157.
7. J. BLOEM, *Philips Res. Rep.* **13** (1958) 167.
8. K. STECKER, *Ann. Physik* **7** (1959) 70.
9. J. DELLACHERIE, Thesis, University of Nancy (1973).
10. A. REVCOLEVSCHI, *Rev. Int. Hautes Temp. et Ref.* **7** (1970) 73.
11. R. D. SCHMIDT-WHITLEY, M. MARTINEZ-CLEMENTE and A. REVCOLEVSCHI, *J. Crystal Growth* **23** (1974) 113.
12. F. A. KROGER and H. J. VINK, "Solid State Physics", Vol. 3, edited by F. Seitz and D. Turnbull (Academic Press, New York, 1956).

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The effect of grain size on the occurrence of cleavage fatigue failure in 316 stainless steel

Highly reflective mirror like cleavage facets occur on the fatigue fracture surfaces of austenitic type 316 stainless steel [1]. The influence of grain size

on this failure mode has been examined for the grain sizes of 3, 20 and 37 grains/mm. Tests at 140 Hz used an Amsler vibrophore and standard compact tension [2] specimens to produce crack propagation in the stress intensity range $\Delta K = 5 - 30 \text{ MN m}^{-3/2}$ at a stress ratio of $R = 0.33$.

A cleavage facet is shown for the 3 grains/mm



Figure 1 Cleavage facet, $\times 44$.

material in Fig. 1. In Fig. 2, % cleavage is plotted against ΔK . The amount of cleavage increases and a peak occurs at higher ΔK values with increase in grain size. Calculation of the stress intensity factor range to give an alternating plastic zone size equal

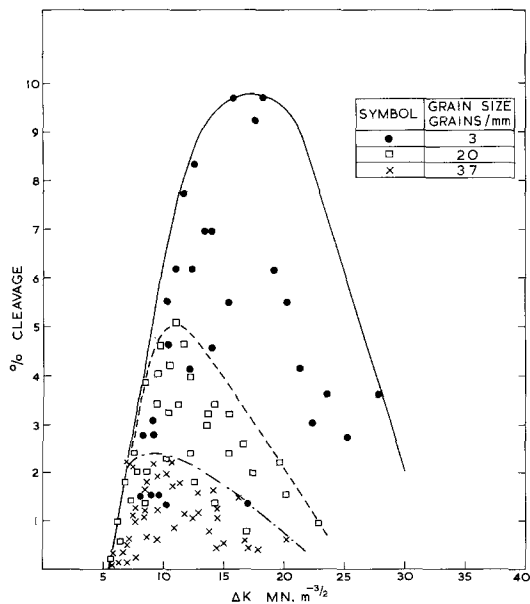


Figure 2 Cleavage fracture as a function of grain size and stress intensity

TABLE I

Grain size (grains/mm)	Stress intensity for peak cleavage	
	Observed	Calculated
37	8	6.8
20	11	9.3
3	18	24

to the grain diameter predicted the approximate values of ΔK at peak % cleavage as in Table I.

Metallographic observations show that the cleavage is associated with reduced plastic deformation in certain grains as evidenced by the reduction in slip lines. The cleavage is also considered to be part of the fatigue process and not isolated areas of fast fracture which occurs in certain ferritic steels [3]. Fig. 3 shows a cleavage facet in which the crack front has been stopped approximately halfway across and marked by a surface "blueing" heat-treatment before testing has continued.

The results shown in Fig. 2 were all produced in laboratory air at ambient temperatures. The occurrence of cleavage was also observed at 500° C in air [1]. Fatigue crack propagation specimens tested by Priddle and Wiltshire [4] in sodium at 500° C were examined but the cleavage failure mode was not observed. Fig. 4 shows scanning

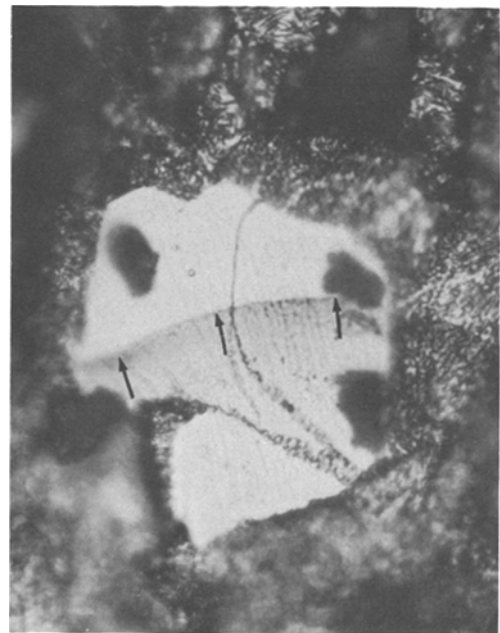


Figure 3 Facet with crack front marked, $\times 800$.

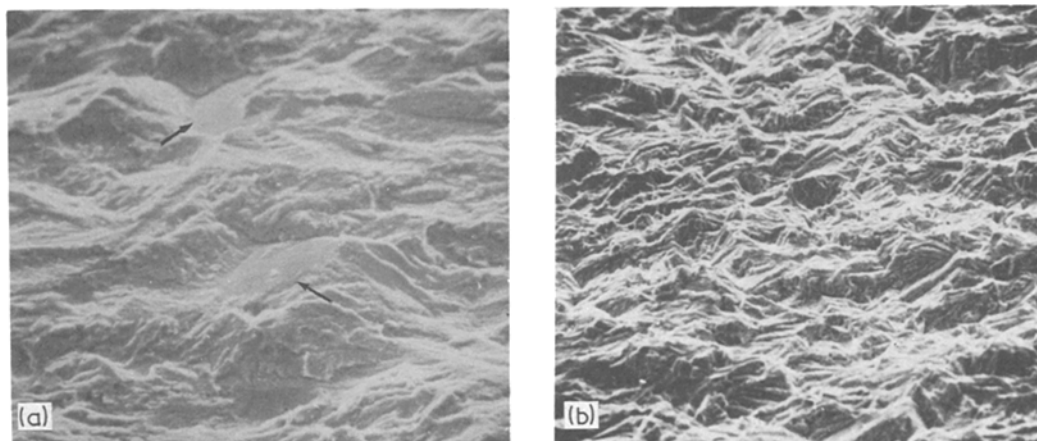


Figure 4 Fracture surfaces of 316 steel, (a) in air at ambient temperature. Two facets are visible; (b) in sodium at 500° C. Facets are not observed. $\times 480$.

electron microscope photographs of the 316 steel fracture surfaces from room temperature and tests at 500° C in sodium.

It is thought that cleavage in fcc materials should occur on the $\{111\}$ planes and this has been observed in stage I fatigue in nickel and aluminium alloys [5]. The faceted fracture areas in the 316 steel of 3 grains/mm size have been examined by X-ray diffraction which has indicated that cleavage does in fact occur on the $\{111\}$ plane.

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References

1. E. K. PRIDDLE and F. E. WALKER, C.E.G.B. Report RD/B/N3384 (in preparation).
2. E. T. WESSEL, *Eng. Fract. Mech.* **1** (1968) 77.
3. R. O. RITCHIE and J. F. KNOTT, *Mat. Sci. Eng.* **14** (1974)
4. E. K. PRIDDLE and C. WILTSHIRE, *Int. J. Fract.*
5. W. J. PLUMBRIDGE and D. A. RYDER, *Met. Rev.* **136** (1969).

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A specimen for evaluating the area of SACPs

Most commercial scanning electron microscopes now offer the capability of obtaining selected-area channelling patterns, SACPs. Different electron optical techniques are used to obtain the SACPs and on the various machines it is difficult to evaluate the minimum area from which the channelling pattern is being generated. In this paper we discuss the preparation of an Al-Ge sample which has proven to be very effective in evaluating the minimum area of SACPs.

An alloy of Al-60wt% Ge is prepared by melting the elements in a 1 in. diameter graphite crucible under an inert atmosphere and then fur-

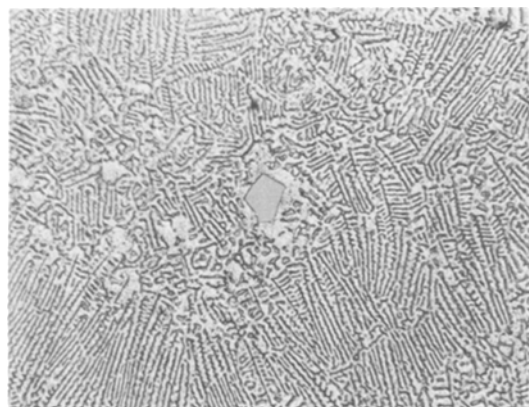


Figure 1 Ge particles in the central region of the ingot. Optical micrograph, $\times 60$.