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J-INTEGRAL TESTING OF ALUMINUM ALLOYS: A NEW TECHNIQUE
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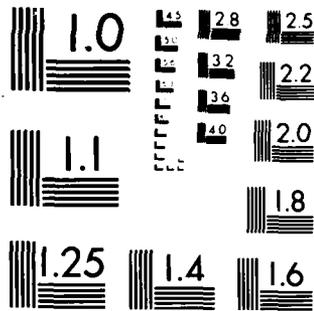
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Structures Technical Memorandum 439

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J-INTEGRAL TESTING OF ALUMINIUM ALLOYS -
A NEW TECHNIQUE FOR MARKING CRACK FRONTS

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SUMMARY

An important aspect of the standardized J_{IC} test method is the measurement of the amount of crack extension as a function of load. The usual method of marking crack fronts for subsequent measurement in aluminium alloys is to fatigue cycle after initial static crack extension.

In this paper, a new technique for marking crack fronts in aluminium alloys is described. This new technique, based on liquid metal embrittlement of grain boundaries in aluminium by gallium, is, for the alloys examined in this work, a more effective method of crack front marking than fatigue cycling.



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1. INTRODUCTION

The J-integral is a fracture mechanics parameter which is used to characterize the elastic-plastic behaviour of engineering materials. In 1972 Begley and Landes [1] first proposed that initial stable crack extension occurs when the value of J reaches a critical value. Since then considerable data, both theoretical and experimental, have been published supporting the use of J_{IC} as an elastic-plastic fracture criterion.

The acceptance of J_{IC} as a measure of the toughness of ductile metals, at or near initial crack extension has resulted in ASTM standardized test methods being developed [2]. An important aspect of these methods is the measurement of the amount of crack extension, Δa , as a function of load. In the single-specimen J_{IC} test method the Δa values are determined indirectly by various methods, as outlined for example in reference [3]. In comparison, for the more frequently used multiple-specimen test method, the Δa values are measured directly from the fracture surfaces of each specimen after the crack front has been marked and the fracture completed. For steels, heat tinting at approximately 300°C for 10 minutes is a good method of marking the crack front after the initial crack extension from the static loading, whereas for other materials, such as aluminium alloys, fatigue cycling at low growth levels is commonly used.

In this paper, a new technique for marking crack fronts in aluminium alloys is described. This new technique, based on the liquid metal embrittlement of grain boundaries in aluminium by gallium, is, for the alloys examined in this work, a more effective method of marking crack fronts than fatigue cycling, as shown for example in Figs 1(a) and (b).

2. BACKGROUND

Grain boundary embrittlement by liquid metals occurs in many solid-liquid systems. A classic example of liquid metal embrittlement is the penetration of gallium along grain boundaries in polycrystalline aluminium. This phenomenon adversely affects the mechanical properties of aluminium and its alloys within a certain temperature range [4]. However, it has been used to advantage in some metallographic investigations of aluminium alloys including the study of grain boundary structures and precipitates [5], and the measurement of sizes and shapes of grains [6].

Intergranular penetration of aluminium by gallium occurs within a temperature range between the melting point of gallium, 29.7°C, and approximately 125°C [4]. Penetration within this temperature range is very rapid and increases with increasing temperature as shown in Table 1. At temperatures above 125°C bulk diffusion occurs and the grain boundary embrittlement by liquid gallium disappears.

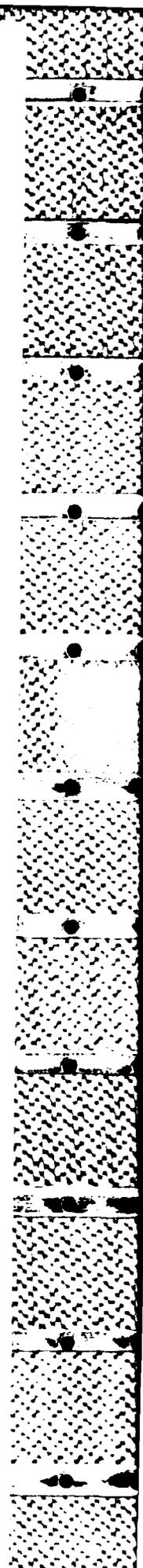
3. EXPERIMENTAL TECHNIQUE

Four high-strength aluminium alloys used in the aircraft industry were tested in this work. The mechanical properties and product forms in which these alloys were tested are given in Table 2. Two specimen geometries, compact tension and centre-cracked tension, with thicknesses between 5 mm and 20 mm, were used to determine the times for liquid gallium to completely diffuse through the thickness.

Demonstration of the new technique for marking the crack fronts in aluminium alloys was as follows:

- 1) The various specimens were fatigue precracked and statically loaded to produce the required amount of stable crack extension as per the ASTM standard.

- II) The surfaces of the specimens were then chemically degreased and polished using silicon-carbide papers down to a 1200 grade finish. This preparation was necessary for two reasons; 1) to allow the liquid gallium to wet the surface in front of the crack tip, and, 2) to make it easier to see when the gallium had completely penetrated through the specimen.
- III) The next step was to preheat the specimens for 15 minutes at 90°C ; this is the temperature used for subsequent grain boundary embrittlement. After preheating, a very small amount of gallium was placed on one of the surfaces of each specimen just in front of the crack tip. Complete through-thickness diffusion of the specimens used in this work was achieved using approximately 0.03 g and 0.08 g of gallium for the 5 mm and 20 mm thick specimens respectively. As the gallium became molten it was worked into the surface in front of the crack tip using a scalpel blade. The scalpel blade was used to scratch the surface below the molten drop to assist the gallium to wet the aluminium.
- IV) The preheated specimens were then placed on a glass plate, the molten gallium was covered with a beaker and heated at 90°C , as shown schematically in Fig. 2. This temperature was chosen because the rate of grain boundary diffusion by gallium is a maximum in the temperature range of $90\text{-}110^{\circ}\text{C}$ [7]. The easiest way to determine when the gallium had completely diffused through any specimen was to examine its bottom surface through the glass plate. Complete diffusion occurred when gallium collected on this surface and only a thin layer of gallium was left on the top surface. If the top surface only had a thin layer of gallium left and there was no sign of any gallium on the bottom surface then an additional amount of gallium was required.
- V) Finally, after the gallium had completely diffused through the specimens the fracture were completed. The force required to break open each specimen was greatly reduced by the grain boundary embrittlement of the remaining uncracked ligament.

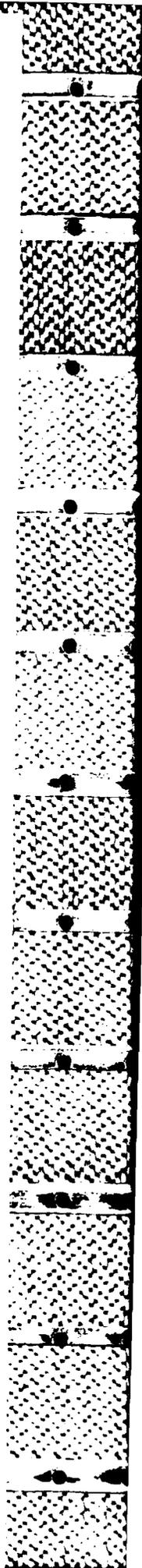


VI) The contrast between the fracture surfaces associated with the fatigue precracking, the static loading, and the final intergranular failure could be further enhanced by leaving the specimens for a few days at room temperature before taking measurements.

4. RESULTS AND DISCUSSION

Crack fronts in the various J-integral specimens, after fatigue precracking and the initial static loading, were marked using both low-amplitude fatigue cycling and the liquid metal embrittlement method. The enhanced contrast between the fracture surface of the static crack extension and that of the final failure, when liquid metal embrittlement is used to mark the crack front instead of fatigue cycling (Fig. 1), is primarily a result of topographical contrast. In general, commercial purity aluminium alloys fail by a predominantly transgranular failure mode during either static [9] or cyclic loading [10]. Consequently the topographical contrast between the static crack extension fracture surface and the final fatigue cycled fracture surface is not great, as shown for example in Figs 3(a) and (b). In comparison, the liquid metal embrittlement of polycrystalline aluminium by gallium produces a much rougher fracture surface which is characteristic of an intergranular mode of failure. Therefore, the contrast between the static crack extension fracture surface and that associated with the liquid metal embrittlement is increased, as shown in Figs 3(a) and (c), compared with the contrast when the crack front is marked by fatigue cycling.

The further improvement in optical contrast with time at room temperature is a result of the reaction of the aluminium with the gallium left on the fracture surfaces in the presence of air. This reaction is responsible for the change in the colour of the fatigue precracked fracture surface and the black products on this surface as shown in Fig. 1(b), compared with the fatigue precracked fracture surface shown in Fig. 1(a).



The times required for liquid gallium to diffuse through the various alloys and specimen geometries at 90°C are given in Table 3. These data cannot be used to determine accurate values for grain boundary diffusion rates of liquid gallium because the rates depend on several factors such as, alloy composition, grain size and texture, and specimen geometry and thickness. However, a rough estimate can be obtained from the following equation [8].

$$C \approx (2.D.t)^{1/2} \quad (1)$$

where C is the distance diffused by the liquid gallium, D is the diffusion coefficient and t is the time. A comparison of the values of D in Tables 1 and 3 shows that the diffusion coefficients for the high strength alloy specimens, with the exception of alloy 2214-T651, were significantly higher than the value for pure aluminium. The general trend could be expected as the grain boundary diffusion of the liquid gallium through surface grains, and hence along a crack front, is faster than through the bulk of the material. The diffusion data for the high purity aluminium in Table 1 were for grain boundary diffusion of the liquid gallium through the bulk of the material.

The variations in the approximate diffusion coefficients for the different alloys and product forms means that it is difficult to estimate the times for complete grain boundary diffusion of liquid gallium for any aluminium alloy or thickness. However, as mentioned in Section 3, the easiest way to determine when complete diffusion has occurred is to observe the bottom face of the specimen through the glass plate.

5. CONCLUSIONS

A new technique for accurately marking crack fronts in aluminium alloys, as required by the ASTM standard multiple-specimen J-Integral test method, has been developed. This technique, based on the liquid metal embrittlement of grain boundaries in aluminium by gallium, is a more effective method of marking crack fronts than the normal method of fatigue cycling, for the alloys examined in this work.



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TABLE 1. Influence of temperature and time on the intergranular diffusion of liquid gallium through 2 mm thick aluminium (99.95% purity) plate [8].

Temperature (°C)	Time for Complete Diffusion (min)	Approximate Diffusion Coefficient D (cm ² /min)
40	10	2.0×10^{-3}
60	9	2.2×10^{-3}
100	7	2.9×10^{-3}
160	> 30	6.7×10^{-4}

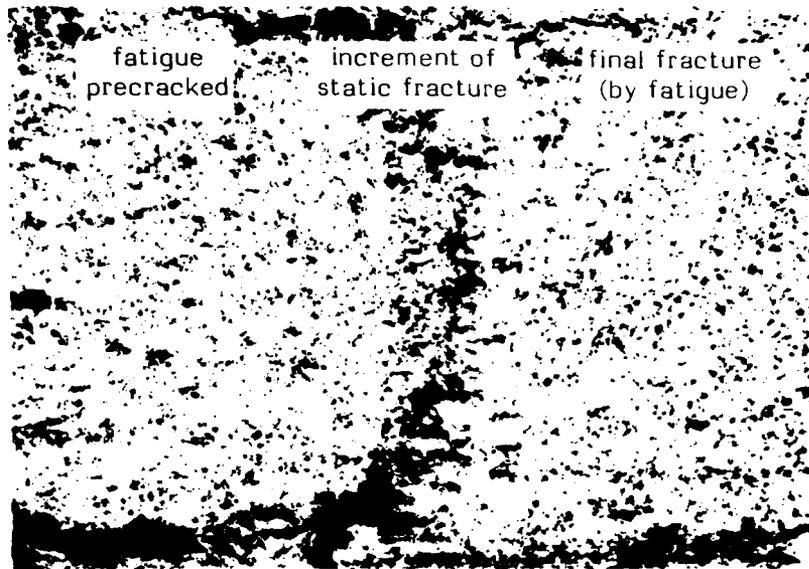
TABLE 2. Mechanical properties and product forms of the aluminium alloy test specimens

Alloy Designation and Temperature	Product Form	0.2% Proof Stress (MPa)	Ultimate Stress (MPa)	Total Elongation %
7475-T7351	wrought	393	469	10.0
7075-T6	forged	463	540	7.0
2024-T351	extruded	426	492	15.8
2214-T651	wrought	390	458	11.5

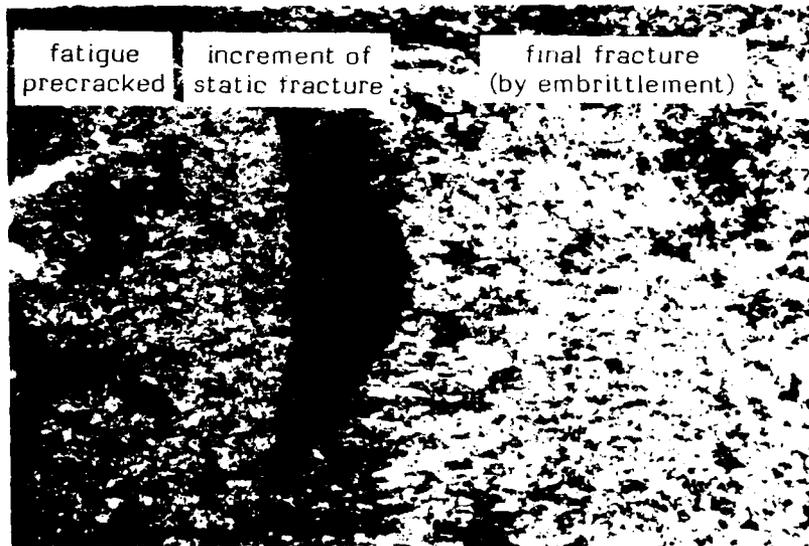
TABLE 3. Intergranular diffusion times of liquid gallium through aluminium alloys at 90°C.

Alloy and Product Form	Specimen* Geometry	Specimen Thickness (mm)	Time for Complete Diffusion (min)	Approximate Diffusion Coefficient $D(\text{cm}^2/\text{min})$
7475-T7351 wrought	CT	5	16	7.8×10^{-3}
7075-T6 forged	CT	20	150	1.3×10^{-2}
2024-T351 extruded	CCT	10	60	8.4×10^{-3}
2214-T651 wrought	CCT	20	770	2.6×10^{-3}

* CT and CCT denote compact-tension and centre-crack tensile specimen geometries respectively.



(a)



(b)

Fig. 1 Marking crack fronts in aluminium alloys after the initial static crack extension using

- (a) fatigue cycling, and
- (b) liquid metal embrittlement by grain boundary diffusion of gallium, for alloy 2024-T351.

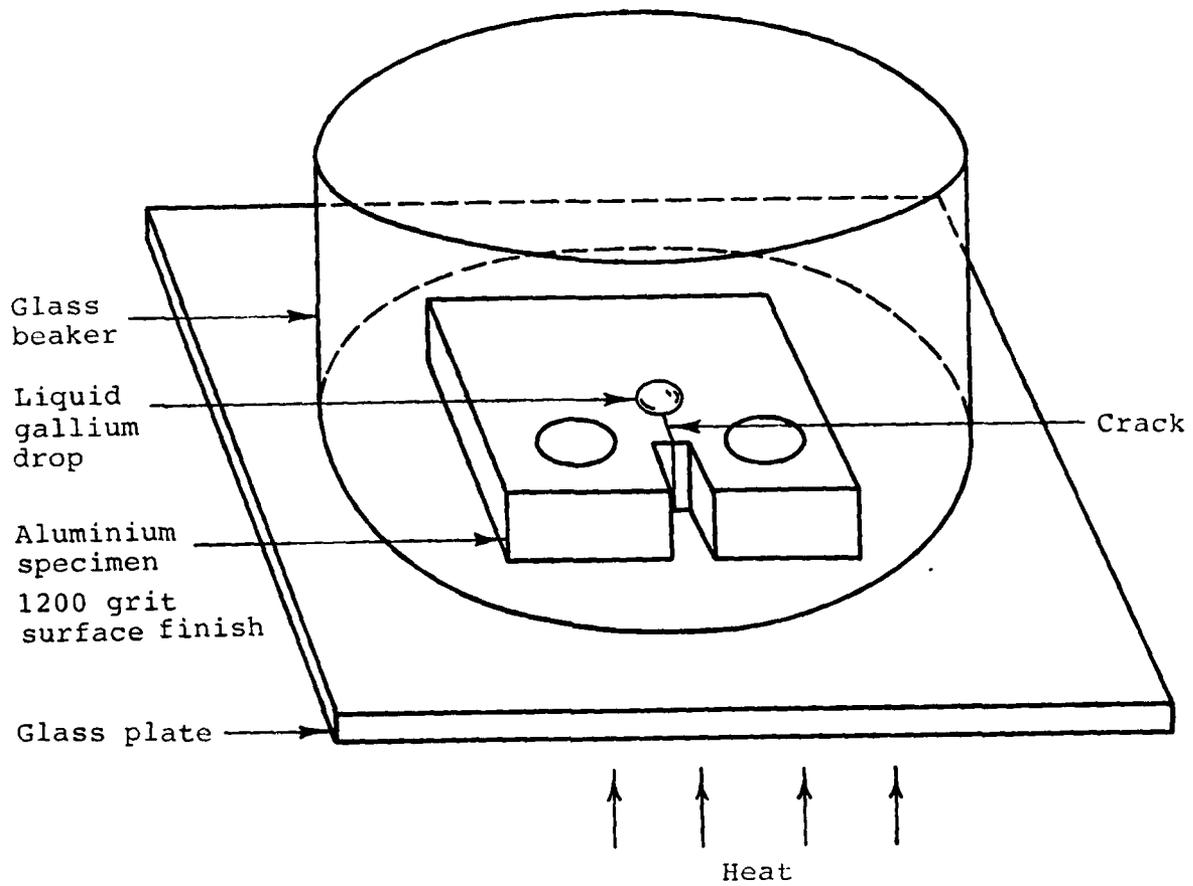
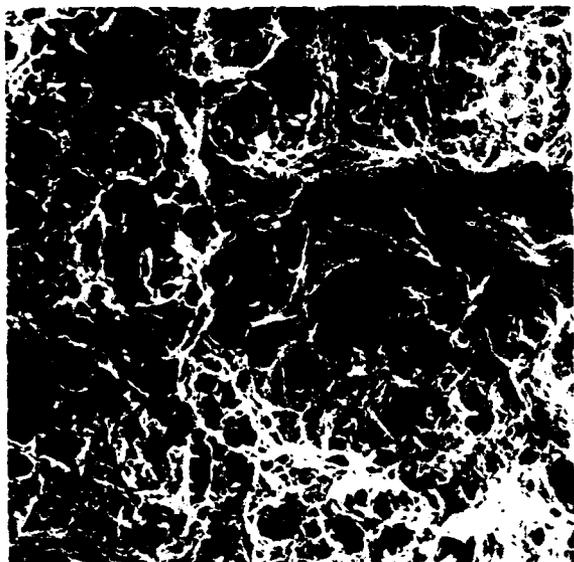
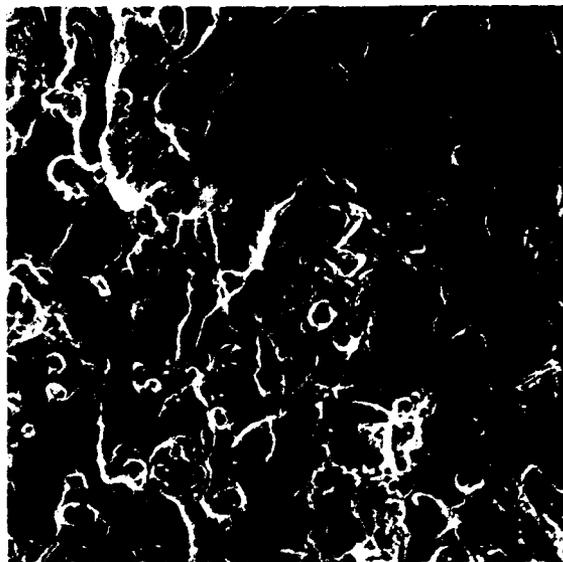


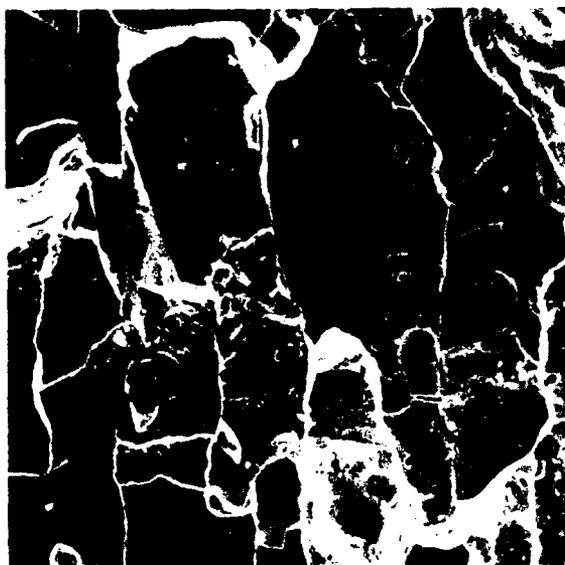
FIG. 2 SCHEMATIC REPRESENTATION OF THE APPARATUS FOR CRACK FRONT MARKING OF ALUMINIUM J-INTEGRAL SPECIMEN USING THE INTERGRANULAR LIQUID METAL EMBRITTLEMENT BY GALLIUM.



(a)



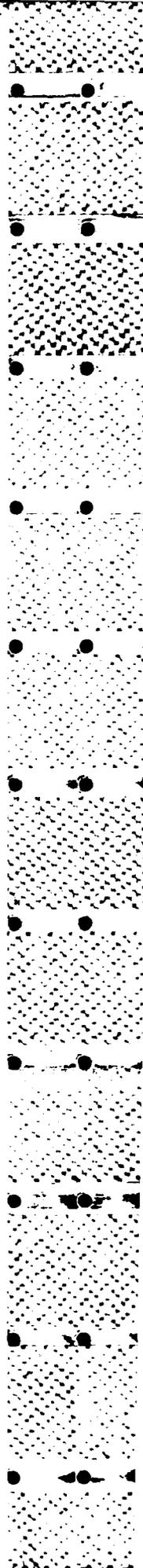
(b)



(c)

Fig. 3. Scanning electron micrographs of the various fracture surfaces for alloy 2024-T351.

- (a) initial static crack extension,
- (b) post fatigue cycling after the initial static loading, and
- (c) intergranular liquid metal embrittlement by gallium after the initial static loading.



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4. Title J-INTEGRAL TESTING OF ALUMINIUM ALLOYS-A NEW TECHNIQUE FOR MARKING CRACK FRONTS		5. Security a. document UNCLASSIFIED	6. No Pages 5
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14. Descriptions Fracture tests Aluminium alloys		15. COSATI Group 11060 11130	
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17. Imprint Aeronautical Research Laboratories, Melbourne		
18. Document Series and Number Structures Technical Memorandum 439	19. Cost Code 256900	20. Type of Report and Period Covered
21. Computer Programs Used		
22. Establishment File Ref(s)		

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