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(6) ACOUSTIC EMISSION
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TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-63-509, Part I

(11) April 1963,

AF Materials Laboratory
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

(17)

(16) Project No. 7360, Task No. 736002

(15)

(Prepared under Contract No. AF33(657)8562,
by Lessells and Associates, Inc., Waltham,
Massachusetts; B.H. Schofield, author).

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**LESSELLS AND
ASSOCIATES INC.
ENGINEERS**

303 Bear Hill Road, Waltham 54, Massachusetts, Telephone: 899-3350-

30 August 1963

Subject: ASD-TDR-63-509
Part I
Contract No. AF 33(657)-8562
Acoustic Emission Under Applied Stress

Gentlemen:

At the request of the Materials Central, Directorate of Advanced Systems Technology, Aeronautical Systems Division, one copy of the subject report is enclosed for your information and retention.

Your comments on this report will be appreciated, and should be forwarded to the Materials Central, Directorate of Advanced Systems Technology, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio, Attention: ASRCM-23 (Mr. H. Kamm). Data contained therein should not be reproduced without permission of the Aeronautical Systems Division.

Yours very truly,
LESSELLS AND ASSOCIATES, INC.



Bradford H. Schofield
Senior Project Engineer

BHS/fb
Enc.

FOREWORD

This report was prepared by B. H. Schofield, Senior Project Engineer, Lessells and Associates, Inc., Waltham, Massachusetts, under USAF Contract No. AF33(657)-8562. This contract was initiated under Project No. 7360, "The Chemistry and Physics of Materials", Task No. 736002, "Nondestructive Methods". The work was administered under the direction of the Metals and Ceramics Division, AF Materials Laboratory, Aeronautical Systems Division, with Mr. H. W. Kamm acting as project engineer.

This report covers work from 15 April 1962 to 15 March 1963.


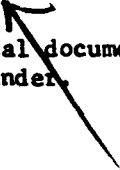
The author wishes to acknowledge the interest and encouragement of Professor Bruce Chalmers, Harvard University. The author is also indebted to Messrs. F. Ranstrom and D. Landers of Lessells and Associates for their assistance in preparing and conducting the experiments.



ABSTRACT

This report concerns specific investigations undertaken to assess the role of the surface oxide layer in the generation of acoustic pulses in metals, i.e., acoustic emission. Experiments were conducted on single crystals of aluminum of different crystallographic orientation. The specimens were pulled in tension both in atmosphere and exposed to etchant solutions. Results demonstrated the oxide layer is not itself an emission source and that the emission characteristics are significantly modified as a result of the influence of the oxide on the deformation behavior of the specimens. Specific observations suggest the operative mechanism of emission generation and the importance of the role played by the surface condition in the deformation of metals.

This technical documentary report has been reviewed and is approved for the Commander.



W. J. TRAPP
Chief, Strength and Dynamics Branch
Metals and Ceramics Division
AF Materials Laboratory

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I. INTRODUCTION

This report describes further studies of the Acoustic Emission phenomenon. Previous reports by the author concerning this research and experimental methods, are noted in References 1 and 2. Other efforts concerning this subject are presented in References 3-12.

Acoustic Emission is the term used to designate the phenomenon of the generation and propagation of acoustic pulses in metals during their deformation. Phenomenological characteristics and historical background of this emission were discussed previously in References 1 and 2. This report is primarily concerned with the specific efforts to determine the relationship of the emission to certain of the deformation characteristics of metals with the ultimate purpose of establishing the source or sources of such emission.

In the preceding research efforts it was demonstrated that the emission is an inherent characteristic of metal deformation intimately related to the fundamental deformation processes. Certain facets of the experimental observations suggested that the surface plays a predominant role, not only with regard to the characteristics, but also the occurrence of the emission, however, the nature of the surface contribution was not clear, i.e., as an influence on the deformation behavior modifying the emission or, as an independent emission source. The experiments described in this report concern this phase of the research.

Relatively few studies have considered the influence of the metal surface on the deformation characteristics of metals. In most cases, with the exception of certain precious metals, e.g., gold and platinum, an oxide layer is present on the surface. According to current hardening theory, the impediments for the movement of dislocations are considered within the material, the barriers due to the surface itself and the existence of solid films being neglected, notwithstanding the fact that considerable evidence demonstrates the surface exerts a large effect. Experiments have shown that the condition of the surface can have a significant effect on the critical shear stress (13,14,15), creep behavior (16,17,18,19), and on the character of the stress-strain curve, i.e., hardening slope, and the suppression of Stage I and at times Stage II regions. Barrett's interesting twist experiments (20,21), demonstrated a paradoxical behavior attributed to the barrier effect of the oxide film. In these tests, when an etchant was suddenly applied to previously twisted single crystals and polycrystalline wires of iron and zinc, a transient condition occurred and the specimen twisted instead of untwisting.

Manuscript released by author April 1963 for publication as an ASD Technical Documentary Report.

Kramer's studies (22,23,24), have also shown several interesting surface effects including transient characteristics which he relates to the kinetics of dislocation behavior at the surface.

A number of mechanisms have been proposed to explain these effects of oxide films. At present, two concepts seem to be most prominent: 1) locking of surface dislocation sources, and 2) pileups due to blocking of dislocations at the surface. Evidence can be presented to rationalize either concept.

It is clear from the preceding, that the surface and its condition has a marked effect on the mechanical behavior of single crystals. Since the emission is intimately related to this mechanical behavior, the emission characteristics would be expected to reflect this influence; the nature of the effect dependent on the peculiarities or the manner in which the mechanisms operate. It is the purpose of this paper to present the changes in the emission behavior for specimens pulled in tension, with and without oxide films, and on which the surface is being removed. Through these observations a better insight into the relation of the emission to deformation mechanisms and the specific nature of the characteristics of these mechanisms may be obtained. The role or contribution of the oxide layer itself will of course also be determined through such experiments.

II. EXPERIMENTAL PROCEDURE

High purity (99.993%) aluminum single crystals, grown from the melt by the Chalmers method, were employed for these studies. The material and specimens were supplied by the Unimet Corporation of Arlington, Massachusetts.

Two forms of specimens were used; the first simply a $\frac{1}{4}$ inch diameter rod specimen four to six inches long, and the second as shown in Figure 1. In this figure, the single crystal portion is shown as the reduced section (3.8 cm long), the remainder of the specimen is a cast high strength aluminum alloy. The simple cylindrical specimens were not designed for load, but were used to study the nature of the etching process itself with regard to generation of acoustic pulses, and for investigations relating to the correlation of deformation and emitted pulses in the etchant solution. Orientations of the single crystal specimens were (100) and 0.5 as determined by back reflection Laue patterns. The stress-strain curves for these orientations are shown in Figure 2.

Figure 3 shows the arrangement employed for simultaneously etching and applying tensile load to the specimen. The split aluminum support plate was lightly clamped around the specimen and the cylindrical glass section placed on this plate. Molten wax was then poured into the glass cylinder, thereby providing support and sealing of the assembly. A methyl alcohol-nitric acid solution placed in the resulting container provided the reagent for polishing and surface removal. In order to confine the etching to the single crystal portion of the specimen, the transition section of the specimen at the upper end was thoroughly coated with wax and the lower end immersed in the wax seal. As shown, a cathode element was also inserted into the container; the specimen acting as anode. A current of 1 to 2 amperes was used during the etching procedure.

The method of loading the specimen has been completely described in previous reports, References 1 and 2. A sketch of this loading system with associated instrumentation is shown in Figure 4. Instrumentation such as emission pickup, amplification, and magnetic tape recording, are identical to that outlined in detail in the above mentioned reports. To repeat briefly, at the end of the specimen is attached an ADP piezoelectric crystal, the output of which passes through a preamplifier stage at a gain of 10^3 , an amplifier at a gain of 10^3 , thence to the magnetic tape recorder at 1 to 1 gain. The lower cutoff frequency of the system is set at 1000 c.p.s. and the upper cutoff at approximately 100,000 c.p.s.

For the tensile test studies the specimen was first etched to assure removal of the transparent oxide film which could, in every case, be observed with oblique light falling away from the specimen. The current was then reduced to zero during the actual specimen loading. Some experiments were also made in which the tensile loading and current were simultaneously applied.

The investigations to assess the acoustic contribution of the etching were made with the specimen completely unloaded.

III. EXPERIMENTAL RESULTS

The emission characteristics for the two orientations 0.5 and (100) were significantly different with the oxide layer removed from the surface. The 0.5 orientation produced essentially no detectable emission during deformations of the order of 20 per cent and only a brief period of emission at the onset of gross yielding. This latter emission was difficult to observe due to the very rapid acceleration to failure caused by local gross yielding. For one 0.5 specimen the etchant solution was removed after approximately 3 per cent deformation, during which no emission was apparent, and allowed to remain in atmosphere for two days. Upon application of load, emission was again apparent, as previously observed in other tests where no etchant had been used. (2)

The (100) orientation produced considerable emission in the etchant solution, but the stress or deformation level at which it became apparent was above that observed on atmospherically tested samples, i.e., the onset of emission was delayed. No apparent differences in the character or form of the emission signals were observed for specimens in atmosphere or in the etchant solution.

Prior to conducting the above experiments, a series of tests were made to assess the possible contribution of the etchant process itself with regard to the generation of acoustic pulses. These tests, on stress free specimens, showed that considerable burst type pulses are produced as the surface is etched away. The rate of acoustic pulse generation was shown to be proportional to the current density with the existence of a lower limit or cutoff current density below which no burst activity was apparent. This lower cutoff appeared reasonably consistent between .4 to .5 amps per square inch. No upper cutoff occurred up to current densities of 2 amps per square inch and no decrease in the proportionality, i.e., no limit approach, could be observed within this range.

At first appearance it was thought that the very active gas bubble formation on the specimen surface was the source of the burst pulses. However, reversal of the current flow, that is changing the polarity of the specimen, resulted in equivalent or greater gas evolution activity on the specimen without any evidence of the burst type pulses except for an initial transient upon application of the reversed current. This transient activity died away quite rapidly and the slight increase in the level of the background noise was attributed to the gas bubble generation. Close observation showed that with the specimen at

either polarity a very slight increase in the background noise level (white noise) was present due to this gas activity on the specimen surface.

Several experiments were made in an attempt to determine whether or not this burst activity, which occurred only during surface removal, was dependent on the degree of deformation of the specimen or simply a normal continuous characteristic of the etching process. These tests were conducted on unloaded specimens. Three cylindrical specimens of (100) orientation were examined; one specimen in the virgin state as received, a second specimen which was pre-strained approximately five per cent in tension, and a third specimen permanently pre-strained in torsion. These specimens were etched in solution at identical current densities.

The test results for the limited number of specimens were somewhat ambiguous, however, there was some qualitative evidence that a correspondence between deformation and acoustic activity existed. The degree of activity for the virgin specimen was less than that for the deformed specimens and all specimens exhibited a decrease in activity as the surface was removed. A part of the ambiguity was related to this latter observation since it was not clear whether the activity decreased due to physical differences within the specimen or a decrease in the chemical potency of the etchant solution. It is expected that this will be clarified in further tests.

The specimen which was pre-strained in torsion was observed optically during the etchant test for evidence of the "twist" phenomenon. No transient twist was observed during any phase of the etching procedure.

In all of these tests a reversal of specimen polarity was made to confirm the burst activity was independent of gas evolution on the specimen surface. Except for the initial transient when the specimen is made cathodic, the burst pulses only appear during surface removal.

Two experiments were undertaken on specimens of (100) orientation with tensile loading and active etching sustained simultaneously. During the loading the current density was varied to observe its influence on the total acoustic emission. Inasmuch as the etching itself produces emission, the previously described tests at zero load were used to establish approximate levels of etchant produced burst activity in order to determine any additional acoustic contribution such as normally observed in tensile tests. Results of these tests showed that the etchant produced bursts and the deformation produced emission could be separately distinguished quite easily since the former is a burst type signal

and the latter the high frequency continuous type emission. Increases in the current density produced corresponding increases in the high frequency emission and conversely, lowering the current density lessened the emission rate and amplitude. It was observed that a sudden increase in current density produced a transient high amplitude period of the emission which rapidly decreased, but to a higher level of emission amplitude than existed at the lower current density. The transient period was not apparent when going from a high current density to a lower value.

It was quite clear in these experiments that the deformation rate and failure could be accelerated by an increase in the current density, the rate being significantly higher than would be expected purely on the basis of the reduction in stress area of the specimen due to the surface removal. This increase in strain rate was reflected in the rapid increase in the amplitude of the high frequency emission spectrum. At stress levels sufficient to sustain an appreciable strain rate that is accelerating toward failure, the current density was reduced to zero, thereby eliminating the etchant produced bursts and permitting clear observation of the high frequency spectrum which increased in amplitude and rate up to specimen failure.

IV. DISCUSSION

The rather unexpected acoustic emission behavior observed for the two single crystal orientations, (100) and 0.5, strongly indicates the relationship of the emission to deformation mechanics, and further, demonstrates the role and contribution of the oxide layer itself.

To consider the latter first: although no significant emission was detected from the 0.5 oxide-free single crystals for rather extensive deformation, emission was abundantly apparent on the oxide-free (100) oriented specimens. It is clear that had the oxide layer been the contributing emission source per se, no signal would have been observed for either type of specimen. The same conclusion can be made with regard to the combined effect of oxide and specimen surface. The fact that the 0.5 orientation had, in previous experiments, produced considerable emission during deformation indicated, therefore, that the role of the oxide coating was significant in its influence on the source mechanism, i.e., effectual rather than causal. The delayed appearance of the emission on the (100) specimen demonstrated further this influential effect of the oxide on the operation of the acoustic mechanism. It is obvious, and has been demonstrated by many others, that deformation is not subdued under such circumstances, but in fact occurs with greater ease at lower stress levels.

The lack of emission during the deformation of the 0.5 specimen and the elimination of signals in the early stages of deformation on the (100) specimens contradicts the tenets proposed in previous reports relating the emission source to the interchange of energy which occurs when a slip step is formed on the surface of the specimen. In that view a portion of the energy was conceived to appear as an elastic wave propagating through the specimen and subsequently detected as an acoustic pulse. Since a high density of slip lines was produced during the subject deformation without acoustic generation such a premise is no longer feasible. On the basis of these observations it is speculated that a negligible amount of the energy associated with the formation of a slip step is dissipated in the form of an elastic wave. Furthermore, these same observations would suggest that, aside from internal barriers, the primary interference with dislocation egress at the surface is that of the oxide layer; the clean surface playing no significant role to inhibit dislocation mobility at the surface. It would be anticipated that for an orientation such as 0.5, during single slip activity, the only effective barrier to dislocation egress would be the oxide layer acting to inhibit the formation of slip steps. At considerably higher degrees of deformation secondary slip systems would come into operation and it is felt that the related dislocation interference to mobility would then predominate internally; the surface condition would not be influential on subsequent behavior.

In the case of the 0.5 oriented specimens it would be expected that single slip would operate to relatively high strains with a similar mode occurring for the early stage of deformation in the (100) orientation. The failure of the 0.5 specimens was invariably localized over a very small volume; this deformation occurring quite suddenly and developing rapidly to failure. Consequently, the deformation of the specimen as a whole was decidedly in the realm of single slip operation. Dislocation mobility in this mode would experience a minimum degree of interference and the dislocations could pass to the surface quite readily. The lack of the effective oxide layer on the surface as a barrier provides a general environment of relative freedom for development of slip activity. The ease with which the specimens deformed and the tendency to exhibit increased creep at load were indicative of the freedom of the deformation mechanisms. At the relatively high stress levels it was obviously more difficult to prevent the specimen deformation from accelerating to failure, particularly for the 0.5 orientation. That is, without the oxide layer, creep was significantly increased over that observed when the oxide coating was intact. In the case of (100) orientation, this behavior was evident in the much lower decay rate of the emission, the inference being that at the sustained stress level the oxide layer greatly retards the egress of dislocations already in motion and the activity decreases very rapidly. With the surface barrier removed, the activity takes considerably longer to cease.

On the basis of the observations in these experiments, it is hypothesized that the emission is related to the unpinning or breaking away of pileups and that a critical size or pileup intensity is necessary for the production of detectable acoustic pulses, that is, a minimum energy level release is required. For the oxide-free 0.5 orientation, a minimum degree of dislocation interference is encountered, single slip probably existing for substantial deformation with very little energy associated with a dislocation pileup. The introduction of a barrier, such as the oxide coating, results in pileups of sufficient energy at breakthrough to produce the acoustic energy.

The very early stages of deformation of the (100) specimens would again be associated with single slip and a minimum of dislocation interference, hence no emission expected. However, at the higher stress levels secondary slip systems are activated, the intersections of which would result in dislocation entanglements and subsequent breaking away as the stress level increased. The occurrence of emission at much lower stress levels with the oxide layer intact would be associated with the blocking and breaking away of the pileups produced during single slip operation. Again, the rise in amplitude of the signals with increasing stress and deformation would be expected as the energy associated with pileups would be greater at the higher stress levels.

The experiments concerned with the zero loading etchant studies also lend credence to the above interpretation regarding emission source. These results, with the specimen both anodic and cathodic, (gas evolution in both modes) suggest that unless a rather marked difference in the dynamics of bubble formation for the two modes exists, the acoustic burst activity is specifically related to the removal of metal from the surface. It is proposed that the surface removal suddenly relieves sites of dislocation pileups and the resulting energy release appears as an acoustic pulse in much the same fashion as similar action under stress when a pileup reaches the "pop-out" stress level. Such a mechanism would certainly appear plausible considering the experiments of both Barrett⁽²⁰⁾ and Kramer⁽²⁴⁾ in which a transient deformation effect is produced by a change in etchant rate. Kramer refers to this mechanism as a dislocation "pop-out" at the specimen surface. The burst form and approximate energy content are identical to the type of burst observed previously on zinc single crystals and 2024 aluminum specimens. It is also significant to note that the transient behavior of the emission, upon changing the specimen from anodic to cathodic polarity, is identical in nature to the creep behavior observed by Andrade and Randall⁽¹⁷⁾ on cadmium crystals under similar environment. The studies of Philips and Thompson⁽¹⁹⁾ present additional information exhibiting effects on creep comparable to those observed on the acoustic effect.

The particular experiments conducted to evaluate the relationship of the emission to dislocation pileups were not entirely conclusive and consequently do not positively verify either the concept of release of pileup energy or the concentration of pileups at the surface. It was also not feasible in the test concluded to assess whether or not the dislocation behavior is controlled by the locking of surface sources or the blocking of dislocations at the surface resulting in pileups.

The slightly higher burst activity of the deformed specimens relative to the virgin crystal and the definite trend toward less burst activity with increasing etch depth do tend to favor the concepts of acoustic energy release upon sudden relief of the barrier and the existence of pileups at the surface barrier. Further studies are, nevertheless, definitely required in this area of effort, in order to establish quantitative correlations with the proposed hypotheses.

The previously mentioned data obtained in the simultaneous etchant-tensile load tests again demonstrated the influence of the surface removal on the emission characteristics, but more important, provided insight into the nature of the emission source and its characteristics. Under these test conditions the high frequency spectrum observed on all specimens tested in atmosphere appeared along with the etchant burst type signal. The total emission was very similar to the

general effect observed on other materials under tension alone, such metals as zinc, 2024 aluminum, tin, and brass. Normally, the pure aluminum single crystals produce only a very small amount of the burst type pulse, the high frequency emission being predominant.

Increases and decreases of current density in accompaniment with increasing load, showed corresponding changes in both the burst rate and high frequency rate and amplitude. In previous work^(1,2), it was shown that the high frequency effect increases in both amplitude and rate of occurrence with increasing strain rate. At the constant load rate, application of higher current density produces a corresponding increase in strain rate due to the reduction in cross sectional area of the specimen. Qualitative tests at different current densities indicates, however, that the increase in emission rate, amplitude, and strain rate, are significantly higher than that expected on the basis of reduction of area alone. That is, the surface removal not only permits operation of the deformation mechanism at lower stress levels, but exposes new dislocation sites at a greater rate.

On the assumption that both the burst type signal and the high frequency pulses both represent the release of energy upon the unpinning of a dislocation site the marked difference in signal characteristic and signal energy suggest a significant difference in the nature of the energy release. In the case of the etchant process, it is hypothesized that the blocked site is relieved to a critical level at which an avalanche process occurs producing a high energy pulse. On the other hand, the high frequency spectrum is associated, in all instances, with an increasing stress level which is the driving force on the dislocation sites. In this case the barrier to mobility is not completely removed and the subsequent dislocation motion is considered to be discontinuous, i.e., step-wise or jerky rather than as an avalanche action. The process would be one of unlocking, pinning, unlocking, etc., under the action of the increasing stress (which itself would exhibit a discontinuous characteristic) such that the total motion consists of a rapid series of small discontinuous jumps. This concept would account for the high frequency spectrum, which in fact, is a succession of rapid pulses. The increase in signal amplitude with stress level is consistent with the higher release energies associated with the higher stress level at which the action occurs.

V. CONCLUSIONS

The oxide coating on the aluminum single crystal specimens is not itself a source of acoustic emission. The unpinning or breaking away of dislocation pileups appears to be the basic source of the emission observed. The results suggest the oxide layer has a significant influence on deformation behavior at the lower stress levels and for single slip activity, while at the higher stress levels and for multiple slip activity, internal barriers appear to be the primary influence on deformation behavior. A critical level of energy is necessary for the generation of a detectable acoustic pulse: the formation of a slip step, per se, does not appear to involve sufficient energy to produce an acoustic pulse. Additional studies are required to verify, quantitatively, the hypotheses proposed.

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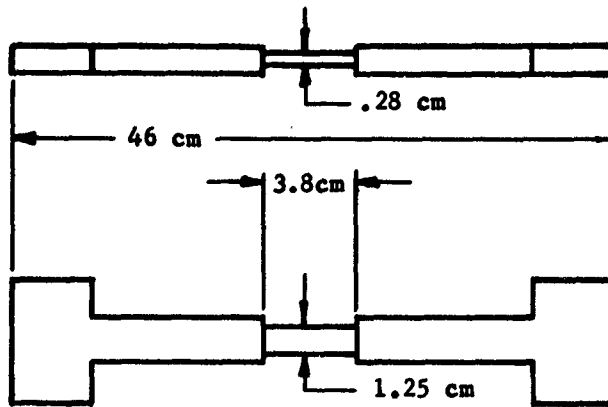


FIGURE 1. Tensile-Etchant Test Specimen

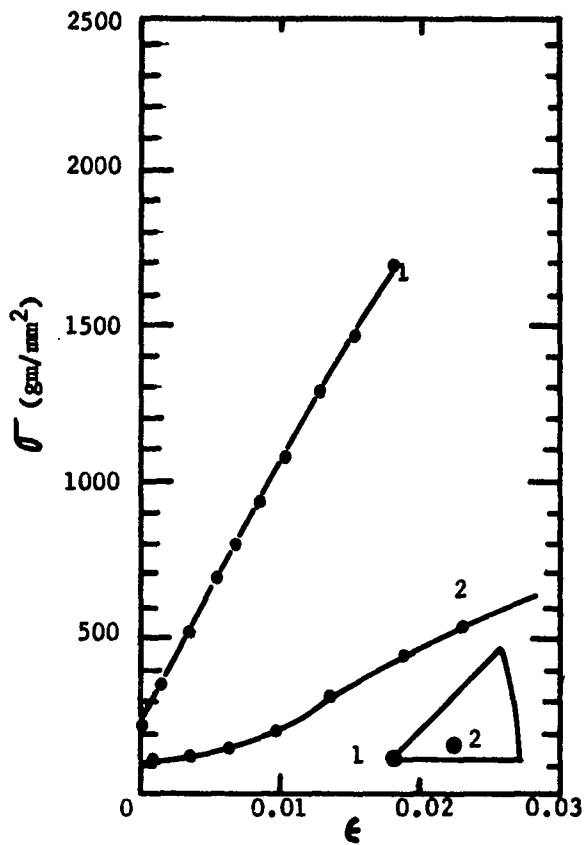
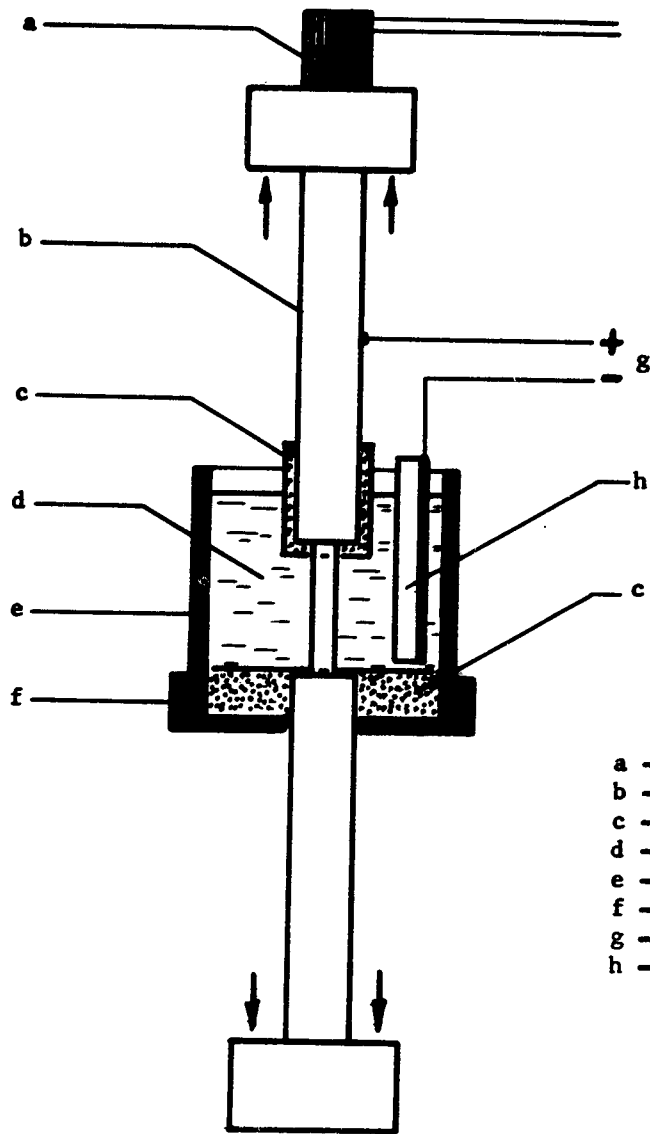


FIGURE 2. Stress-Strain Curves and Axial Orientations of Aluminum Single Crystal Test Specimens



- a - piezoelectric transducer
- b - specimen (anode)
- c - wax coating seal
- d - etchant bath
- e - glass sleeve
- f - split support
- g - battery
- h - cathode

FIGURE 3. Specimen Arrangement for Simultaneous Tensile-Etchant Testing

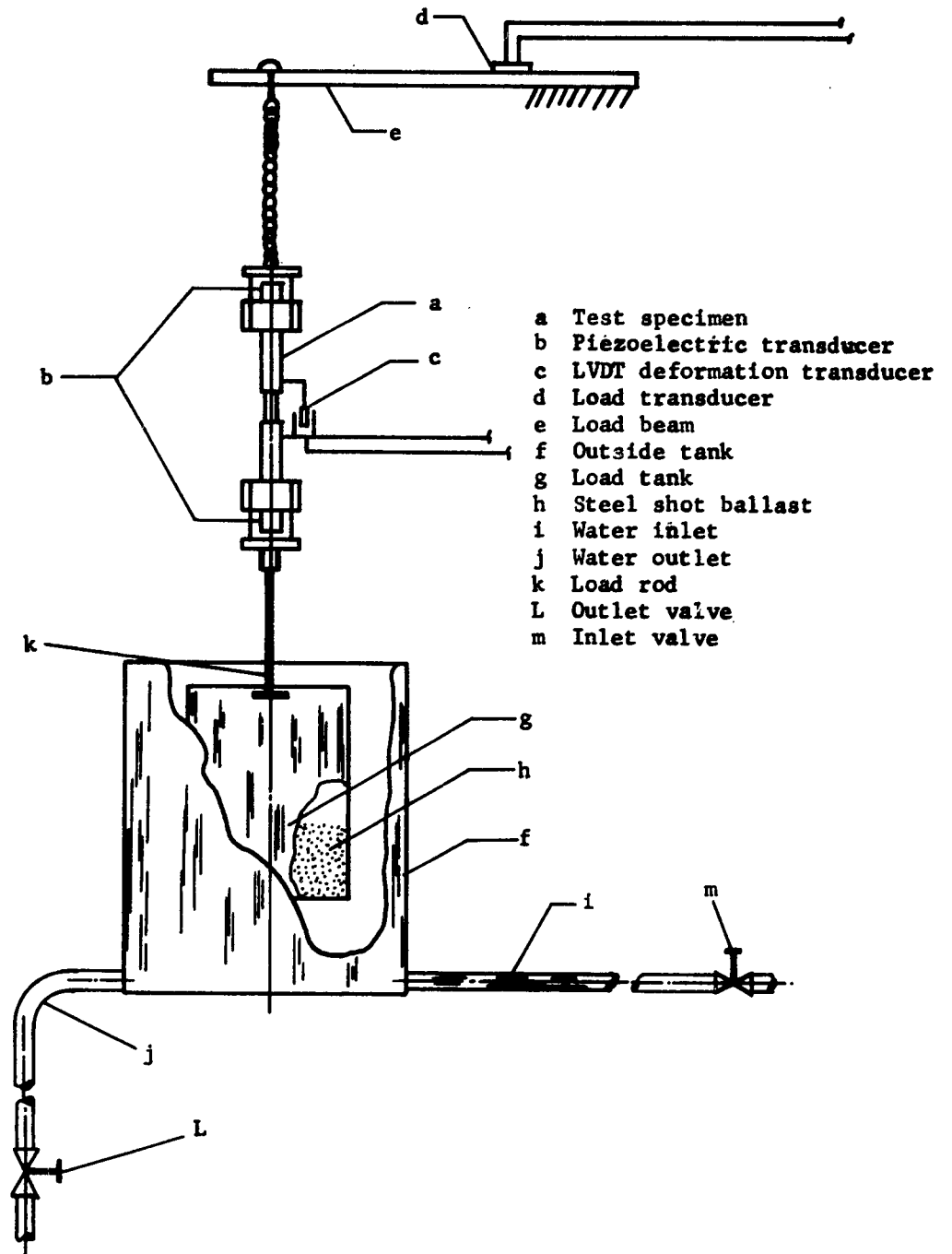


FIGURE 4. Test Apparatus
17

1. Deformation of Metals
2. Metallurgy (Mechanical)
3. Acoustics
4. Single Crystals
5. Surface Effects
- I. AFSC Project 7360, Task 736002
- II. Contract AF33(657)-3562
- III. Lessells and Associates, Inc.
- IV. B.H. Schofield
- V. Aval fr OTS
- VI. In ASTIA collection

Aeronautical Systems Division, AF Materials Laboratory, Metals and Ceramics Division, Wright-Patterson AFB, Ohio.
Rpt. No. ASD-TDR-63-509, Pt. I. ACOUSTIC EMISSION UNDER APPLIED STRESS. Interim report, June 1963, 17pp. incl illus, 24 refs.

Unclassified Report

This report concerns specific investigations undertaken to assess the role of the surface oxide layer in the generation of acoustic pulses in metals, i.e., acoustic emission. Experiments were conducted on single crystals of aluminum of different crystallographic orientation. The specimens

were pulled in tension both in atmosphere and exposed to etchant solutions. Results demonstrated the oxide layer is not itself an emission source and that the emission characteristics are significantly modified as a result of the influence of the oxide on the deformation behavior of the specimens. Specific observations suggest the operative mechanism of emission generation and the importance of the role played by the surface condition in the deformation of metals.

Aeronautical Systems Division, AF Materials Laboratory, Metals and Ceramics Division, Wright-Patterson AFB, Ohio.
Rpt. No. ASD-TDR-63-509, Pt. I. ACOUSTIC EMISSION UNDER APPLIED STRESS. Interim report, June 1963, 17pp. incl illus, 24 refs.

Unclassified Report

This report concerns specific investigations undertaken to assess the role of the surface oxide layer in the generation of acoustic pulses in metals, i.e., acoustic emission. Experiments were conducted on single crystals of aluminum of different crystallographic orientation. The specimens

were pulled in tension both in atmosphere and exposed to etchant solutions. Results demonstrated the oxide layer is not itself an emission source and that the emission characteristics are significantly modified as a result of the influence of the oxide on the deformation behavior of the specimens. Specific observations suggest the operative mechanism of emission generation and the importance of the role played by the surface condition in the deformation of metals.

1. Deformation of Metals
2. Metallurgy (Mechanical)
3. Acoustics
4. Single Crystals
5. Surface Effects
- I. AFSC Project 7360, Task 736002
- II. Contract AF33(657)-8562
- III. Lessells and Associates, Inc.
- IV. B.H. Schofield
- V. Aval fr OTS
- VI. In ASTIA collection