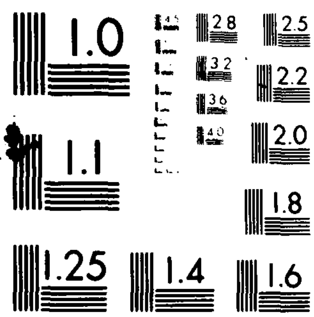
AD-A110 057 MATERIALS RESEARCH LABS ASCOT VALE (AUSTRALIA) F/G 20/11
THE INFLUENCE OF GRAIN BOUNDARY SULPHIDES ON HOT DUCTILITY,(U)
OCT 81 R C ANDREW, G M WESTON

UNCLASSIFIED MRL-R-832 NL

END
DATE
FILMED
2 82
DTIC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

MRL-R-832

AR-002-738

R

AD A110057

DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

MATERIALS RESEARCH LABORATORIES

MELBOURNE, VICTORIA

REPORT

MRL-R-832

THE INFLUENCE OF GRAIN BOUNDARY SULPHIDES ON HOT DUCTILITY

R.C. Andrew and G.M. Weston

Approved for Public Release

DTIC FILE COPY

A

OCTOBER, 1981

82 01 26 045

DEPARTMENT OF DEFENCE

MATERIALS RESEARCH LABORATORIES

REPORT

MRL-R-832

THE INFLUENCE OF GRAIN BOUNDARY SULPHIDES ON HOT DUCTILITY

R.C. Andrew and G.M. Weston

ABSTRACT

DEG

The effect grain boundary sulphide precipitation on the hot ductility of a low-alloy ESR steel has been investigated within the temperature range 1000-1350°C. It was found that grain boundary sulphides on their own have little influence on hot ductility. However, longer exposure times at high temperatures, in the presence of a partially oxidizing atmosphere caused a considerable drop in ductility for test temperatures below 1200°C. The mechanism involving this loss in ductility was not determined.

Approved for Public Release

POSTAL ADDRESS: Chief Superintendent, Materials Research Laboratories
P.O. Box 50, Ascot Vale, Victoria 3032, Australia

DOCUMENT CONTROL DATA SHEET

Security classification of this page: UNCLASSIFIED

1. DOCUMENT NUMBERS:	**2. SECURITY CLASSIFICATION:**
a. AR Number: AR-002-738	a. Complete document: UNCLASSIFIED
b. Series & Number: REPORT	b. Title in isolation: UNCLASSIFIED
c. Report Number: MRL-R-832	c. Abstract in isolation: UNCLASSIFIED

3. TITLE:

THE INFLUENCE OF GRAIN BOUNDARY SULPHIDES ON HOT DUCTILITY

4. PERSONAL AUTHOR(S): ANDREW, R.C. and WESTON, G.M.	**5. DOCUMENT DATE:** OCTOBER, 1981
	6. TYPE OF REPORT & PERIOD COVERED: REPORT
7. CORPORATE AUTHOR(S): Materials Research Laboratories	**8. REFERENCE NUMBERS:** a. Task: DST 77/062 b. Sponsoring Agency:
	9. COST CODE: 54-4840
10. IMPRINT (Publishing establishment) Materials Research Laboratories P.O. Box 50, Ascot Vale, Vic. 3032 OCTOBER, 1981	**11. COMPUTER PROGRAMME(S):** (Title(s) and language(s)):

12. RELEASE LIMITATIONS (of the document):

Approved for Public Release

12-0. OVERSEAS: N.O. [ ] P.R. [1] A [ ] B [ ] C [ ] D [ ] E [ ]

13. ANNOUNCEMENT LIMITATIONS (of the information on this page):

No Limitation

14. DESCRIPTORS:

Ductility
Low Alloy Steels
Electroslag Refined Steels
Hot Ductility
Grain Boundary
Sulphide Precipitation

15. COSATI CODES: 1106

16. ABSTRACT (if this is security classified, the announcement of this report will be similarly classified):

The effect grain boundary sulphide precipitation on the hot ductility of a low-alloy ESR steel has been investigated within the temperature range 1000-1350°C. It was found that grain boundary sulphides on their own have little influence on hot ductility. However, longer exposure times at high temperatures, in the presence of a partially oxidizing atmosphere caused a considerable drop in ductility for test temperatures below 1200°C. The mechanism involving this loss in ductility was not determined.

CONTENTS

THE INFLUENCE OF GRAIN BOUNDARY SULPHIDES ON HOT DUCTILITY

1. INTRODUCTION

The different microstructural changes which may occur when a steel is heated at temperatures high in the austenite range (e.g., prior to a hot-working operation) have recently been described by Samuels[1]. Two such altered structures which have been the subject of numerous investigations are grain boundary sulphide precipitation and grain boundary liquation [1,2], colloquially known as overheating and burning respectively. Much is now known about the conditions which lead to their occurrence and also the underlying mechanism [3,4].

In low-sulphur steels, particularly those produced by special remelting processes such as electroslag refining (ESR) and vacuum arc remelting (VAR), the precipitation of small α-MnS particles on high-temperature austenite grain boundaries may be detected after heating at temperatures as much as 300°C below those at which grain boundary liquation is first observed [3]. Thus, temperatures which lead to extensive precipitation may lie well within the normal hot-working range. Consequently, concern has been expressed that the performance of hot-worked components made from remelted steel may be impaired by extensive solid-state precipitation of sulphides. Accordingly, considerable effort has recently been directed to defining its influence on a wide range of mechanical properites [5-7], generally at room temperature.

By contrast, information about the effect of grain boundary sulphides on hot ductility, in isolation from other high temperature phenomena such as liquation, is not available even though ingot break-up during hot working is still a common event.

As components which have failed to survive either a hot-working operation or a non-destructive inspection after hot-working are periodically submitted to MRL for examination, work was undertaken with the aim of identifying the influence that this major high temperature phenomena has on hot ductility.

The hot impact tensile test was selected as the most appropriate test procedure for determining hot ductility, after discussions with staff of the BHP Melbourne Research Laboratories (BHP MRL) where it has been used extensively in numerous studies on the behaviour of a wide range of steels at elevated temperature.

2. EXPERIMENTAL PROCEDURE

Twenty-one oversize blanks, approximately 20 mm square by 95 mm long, were cut from a segment of a gun barrel which had been forged from an electroslag refined ingot of low-sulphur, low-alloy steel. Portions of this forging have been used previously for studies of the grain boundary sulphide precipitation phenomenon [7,8]. The composition of this steel is given in Table I. The longitudinal axis of each blank was aligned parallel to the original hot-working operation. All blanks were then accurately machined to the dimensions of the hot impact tensile test-piece shown in Fig. 1.

The main feature of this test-piece design is a short parallel gauge section of length 4.76 mm and diameter 6.35 mm. This short gauge length is found to give ductility measurements which are more discriminating than from the usual test-piece, possibly because the reduced gauge length induces necking at lower strains and tends to promote fracture at inclusions [9]. The test-pieces were divided into three groups based on different heat-treatment cycles.

Group A: Heated to temperatures in the range 1000-1350°C, held for ten minutes at temperature, and then fractured.

Group B1: Heated for 10 minutes at 1350°, cooled at a rate of 15-25°C/min to temperature in the range 1000-1300°C (known to produce extensive grain boundary sulphide precipitation) [8], held for ten minutes at the second temperature, and then fractured.

B2: Specimens with their gauge length chromium plated followed the above heat-treatment cycle.

B3: The above heat-treatment time cycle was shortened by reducing the holding times at 1300°C and the test temperature to two minutes.

Group C: Heated at 1400°C for one hour under an inert argon atmosphere*, and cooled to room temperature to produce extensive grain boundary

*This part of the heat-treatment was done at MRL.

sulphide precipitation [8]. Reheated to temperatures in the range 1100-1350°C, held for ten minutes at temperature, and then fractured.

Hot ductility tests were conducted by heating specimens to the desired temperatures in a spilt, resistance-wound furnace mounted on a modified creep machine [9]. The test temperature was measured by a Pt/Pt + 13%Rh thermocouple attached to the gauge length. Test-pieces were fractured in tension by releasing a counter-weighted arm attached to the specimen head. Under these conditions, a strain rate of approximately 10 sec^{-1} was achieved, this being similar to that prevailing under practical rolling conditions, and close to that where the fracture mechanism is largely determined by inclusions [10]. Although the furnace could not be adequately sealed from the surrounding atmosphere, a crude protective atmosphere was maintained by bleeding argon through a hole in the furnace wall and blocking the openings at each end with "Kaowool".

After fracture the test-pieces were rapidly quenched into water at room temperature, the time interval from fracture to quenching being about 1 minute. The reduction in diameter of the parallel gauge length, measured on a shadowgraph, was used as a measure of the hot ductility of each test-piece. The test-pieces were subsequently sectioned longitudinally, polished and then etched to enable the extent of grain boundary sulphide precipitation to be determined [8].

Because of the high test temperatures and the considerable delay before quenching the fractured test-pieces, little useful information was gained from examination of the fracture surfaces.

3. RESULTS AND DISCUSSION

The results of all hot impact tensile tests are summarised in Table II, and in graphical form in Fig. 2. As the heating and testing procedure for group A test-pieces prevented the formation of grain boundary sulphides, their ductility values provide a base level against which to compare those test-pieces in Groups B and C in which varying degrees of sulphide precipitation were produced prior to fracture.

The increase in ductility with increasing test temperature evident in the Group A results follows a pattern similar to that observed for a wide range of steels [11]. This increase is associated with a decline in the hot strength of low-alloy austenites at temperatures in excess of 0.6 of their melting point [12]. Another feature of the Group A results was the overall high level of ductility in the temperature range examined. Gittins and Hinton [11] have proposed three bands of ductility based on the degree of reduction in diameter:

Band I - high ductility, reduction in diameter > 60%

Band II - intermediate ductility, reduction in diameter 40-60%

Band III - low ductility, reduction in diameter < 40%

The results for the Group A test-pieces lie at the top of Band I, confirming forge plant experience that this steel can be easily worked over a wide range of temperatures.

The results determined for test-pieces heated at 1350°C and then cooled to the test temperature (Group B1) were similar to those of Group A when tested at temperatures down to 1200°C. Below this temperature however, ductility values were much lower than the corresponding ones of Group A; for example the value at 1100°C was 50% compared with 85% (Table II, Fig. 2).

Examination of the longitudinal sections of Group B test-pieces revealed that the amount of manganese sulphide precipitation on the high temperature austenite grain boundaries was extensive and uniform at the lower test temperatures Fig. 3. In practice, however, such sulphide networks as outlined by the severe sulphide selective etch in (Fig. 3), would not usually be observed at room temperature. This is because hot-working operations in practice are normally continued down to temperatures approaching 900°C, and therefore, such networks are effectively broken-up on forming. The degree of surface oxidation also increased with decreasing test temperature, being much more severe than previously observed on Group A test-pieces fractured at the same temperature. The opening up of grain boundaries along the surface of both the gauge length and adjoining barrel sections was also a feature at lower test temperatures (Fig. 4). Similar boundary openings were not observed in Group A samples.

The initial intension of the Group B1 heating cycle was to follow closely that experienced in forge shop practice. One practical problem, however, was that the times of exposure to high temperature for all of Group B1 test-pieces were much longer than for Group A. This could amount to as much as twenty minutes for specimens fractured below 1200°C, in conditions where a partially oxidizing furnace atmosphere was unavoidable. Therefore, diffusion of elements such as oxygen and nitrogen along grain boundaries*, may have contributed to the lower ductility values recorded for Group B1 test-pieces fractured below 1200°C. A further consideration here is the small specimen diameter; it is well established [13] that, for a given exposure time under reactive furnace conditions, the smaller the specimen size the greater the reduction in mechanical properties. In large forgings, such surface reactions remain totally unnoticed.

---

* For the sample in question, the possibility that the lower ductility values may have resulted from reactions involving both the migrating species and grain boundary sulphides cannot be overlooked. However, because of the destruction of fracture surfaces by heavy oxidation, this aspect is outside the scope of the present report.

To offset this additional exposure time, the gauge lengths of two test-pieces were chromium plated (B2), and a third (B3, unplated), had its high temperature cycle time shortened. The ductility of plated test-pieces was again low, approximately 41% at 1100°C and 52% at 1000°C. On examination of longitudinal sections, the surface chromium layer was found to be imperfect, a feature not unexpected with this test-piece geometry. The discontinuities in the plating often coincided with grain boundary openings similar to those observed previously in unplated samples, effectively negating the apparent protection afforded by the plating. For the unplated test-piece where the heating cycle was reduced by approximately 15 minutes, the ductility value of 73% at 1100°C represented a substantial improvment over those earlier values with the standard heating cycle. No surface grain boundary openings were observed along the gauge length, although uniform sulphide networks were still observed throughout the sample. As the ductility value here approached the values obtained in Group A samples, high-temperature exposure times, rather than grain boundary sulphides, appeared to be the major factor in lowering ductility.

To enable furnace heating times to be minimized, grain boundary sulphide precipitation in Group C test-pieces was induced in a separate heat-treatment where oxygen potentials could be maintained at very low levels. Under these circumstances a Group A test-procedure could be used, enabling any grain boundary sulphide influence to be identified. The high-temperature ductility values for these Group C test-pieces (Table II, Fig. 2) were nearly identical with those values recorded for Group A tests.

The high ductility levels above were obtained in spite of the presence of heavy grain boundary sulphide networks. This suggests that precipitation by itself has little influence on high temperature ductility and therefore, would not lead to ingot break-up during forging operations.

4. CONCLUSIONS

Extensive grain boundary sulphide precipitation has been found to have negligible effect on the hot ductility of a low sulphur, low alloy ESR steel.

The hot ductility of the steel below 1100°C was substantially reduced after prolonged heating under oxidizing conditions. The mechanism by which this oxidation reduces ductility and the degree of its dependence on the presence of grain boundary sulphides is not clearly understood and could form a basis for futher work.

5. ACKNOWLEDGEMENTS

The authors wish to thank the Director, Melbourne Research Laboratories, BHP for permitting access to the hot ductility equipment and Dr G. Glover, Dr P. Lloyd and Mr M. Toyama of these Laboratories for assisting with the planning and execution of the test programme.

6. REFERENCES

1. Samuels, L.E. "Optical Microscopy of Carbon Steels", American Society for Metals, 1980, p257.

2. Andrew, R.C. and Weston, G.M., Metals Australasia, 1980, 12, 3, 7.

3. Bodimeade, A.H., O'Brien, R.N., Jack, D.H. and Nutting, J. "The Influence of Overheating Upon the Toughness of Heat-Treated Low Alloy Steels", Paper 17, Conference on "Inclusions and Their Effects on Steel Properties", British Steel Corporation, Leeds, 1974.

4. Glue, D.R., Jones, C.H. and Lloyd, H.K.M., Metals Technology, 1975, 2, September, 416.

5. Lake, R.J. "Overheating Characteristics of Clean Gun Steels", RARDE Technical Report 13/75, December, 1975.

6. Gardiner, R.W. Metals Technology, 1977, 4, December, p. 536.

7. Andrew, R.C., Weston, G.M. and Ritter, J.C. Metal Science, 1979, 13, February, p 78.

8. Andrew, R.C. and Weston, G.M. J. Aust. Inst. of Metals, 1977, 22, p 271.

9. Gittins, A., Sellars, C.M. and Northway, L.G. BHP Melb. Res. Labs. Report MRL 29/2, August 1970 and J. of Materials, 1970, 7, 2, p 155.

10. Gittins, A., Hinton, L.G. and Tegart, W.J. McG. Manuf. Eng. Trans., 1973, 2, p 199.

11. Gittins, A. and Hinton, L.G. "Hot Workability Atlas", BHP Melb. Res. Labs. Report S3/16, January, 1973.

12. Tegart, W.J. McG. and Gittins, A. "The Hot Deformation of Austenite" ed. by J.B. Ballance, The Metallurgical Society, 1977, pp 1-46.

13. Nicoll, A.R. and Hildebrandt, U.W. "Thermal Fatigue of Coated Superalloys", Conference on "Environmental Degradation of High Temperature Materials", The Institute of Metallurgists, London, March 1980, 2, 6/27.

TABLE I

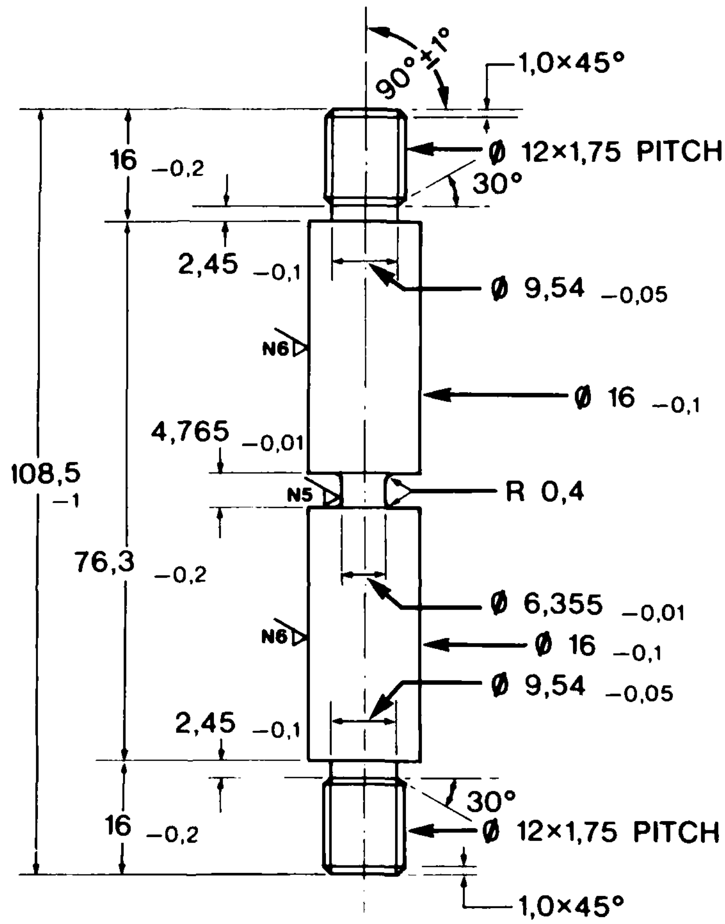
COMPOSITION OF LOW-ALLOY ESR STEEL, WT-%

C	S	P	Si	Mn	Ni	Cr	Mo	V
0.36	0.005	0.008	0.26	0.54	3.1	0.79	0.65	0.19

TABLE II

RESULTS OF THE HOT IMPACT TENSILE TESTS

Heating Cycle	Presence of Grain Boundary Sulphides	Per Cent Reduction in Diameter at Temperatures Shown					
		1000°C	1100°C	1200°C	1250°C	1300°C	1350°C
GROUP A Heated to test temperature and fractured.	No	79	85	93	89	96	95
GROUP B Heated to 1350°C slow cooled to test temperature and fractured.	Yes		50	89	94	96	97
B2 As above, chromium Plated gauge length.		52	41				
B3 As above, except heating cycle shortened markedly.			73				
GROUP C Pre-heat treated at 1400°C, under argon, then treated as for Group-A.	Yes		86	92	93	94	95



NOTE: Dimensions in millimeters.

FIG. 1 - Hot impact Tensile Test-Piece

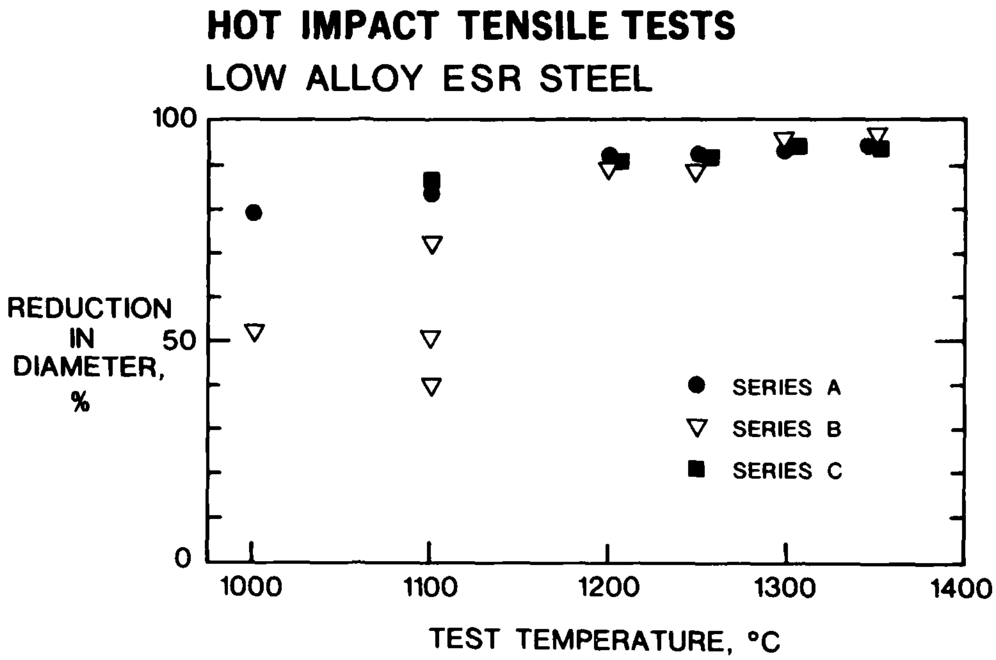


FIG. 2 - Influence of tensile test temperatures on the following samples:
Series A, heated directly to test-temperature and fractured.
Series B, heated directly to 1350°C, then slow cooled to test-temperature and fractured.
Series C, Pre-heattreated at 1400°C, under argon then reheated as for Series A.

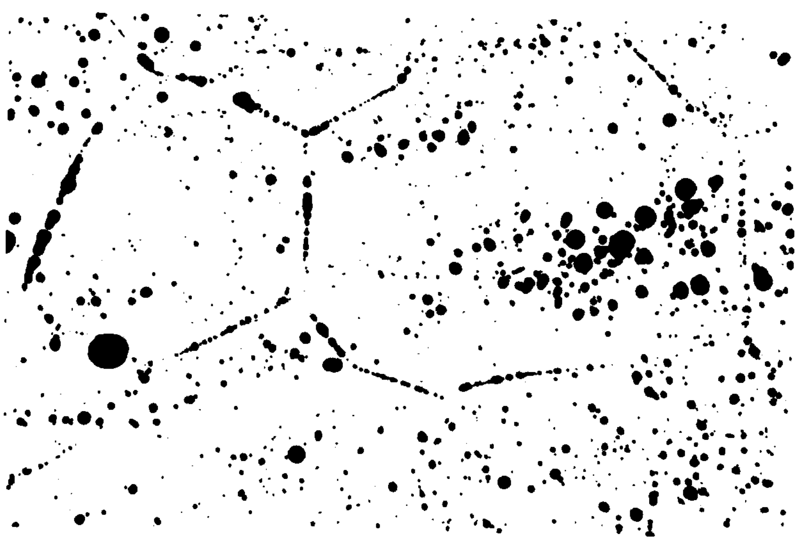


FIG. 3 - Longitudinal section of a group-B specimen fractured at 1100°C showing extensive grain boundary sulphide precipitation. X50

(a)

(b)

FIG. 4 - (a) Polished longitudinal section through half of a group B specimen fractured at 1100°C, showing the characteristic grain boundary openings associated with low ductility test-pieces. X20

(b) An area of the above section showing heavy surface oxidation and cracking along grain boundaries on which sulphide precipitation was present X50 etched in 10 H_2SO_4/10 HNO_3 Solution.

DISTRIBUTION LIST

MATERIALS RESEARCH LABORATORIES

Chief Superintendent
Superintendent, Metallurgy Division
Dr R.C. Ritter
Library
Mr. R.C. Andrew
Mr. G.M. Weston

DEPARTMENT OF DEFENCE

Chief Defence Scientist/Deputy Chief Defence Scientist/
    Controller, Projects and Analytical Studies — 1 Copy
Superintendent, Science and Technology Programme
Controller, Service Laboratories and Trials
Army Scientific Adviser
Air Force Scientific Adviser
Navy Scientific Adviser
Librarian, (Through Officer-in-Charge), Materials Testing
    Laboratories, ALEXANDRIA, N.S.W.
Senior Librarian, Aeronautical Research Labortories
Senior Librarian, Defence Research Centre Salisbury, S.A.
Officer-in-Charge, Document Exchange Centre, — (17 copies)
Technical Reports Centre, Defence Central Library
Central Office, Directorate of Quality Assurance -
    Air Force
Deputy Director Scientific and Technical Intelligence,
    Joint Intelligence Organisation.
Librarian, Bridges Library
Defence Scientific and Technical Representative,
    Australia High Commission, London — (Summary Sheets only)
Counsellor Defence Science, (Attn: CONDS) — (Summary Sheets only)

DEPARTMENT OF INDUSTRY AND COMMERCE

NASA Canberra Office, Woden, A.C.T.
Head of Staff, British Defence Research and Supply
    Staff (Aust.)

DISTRIBUTION LIST

(Continued)

OTHER FEDERAL AND STATE DEPARTMENTS AND INSTRUMENTALITIES

The Chief Librarian, Central Library, C.S.I.R.O.
Library, Australian Atomic Energy Commission Research Establishment

MISCELLANEOUS - OVERSEAS

Reports Centre, Directorate of Materials Aviation, England
Library - Exchange Desk, National of Bureau of Standards, U.S.A.
UK/USA/CAN/NZ ABCA Armies Standardization Representative (4 copies)
The Director, Defence Scientific Information and Documentation Centre, India
Military, Naval and Air Adviser, High Commission of India, Canberra
Director, Defence Research Centre, Kuala Lumpur, Malaysia
Exchange Section, British Library, U.K.
Periodicals Recording Section, Science Reference Library, British Library, U.K.
Library, Chemical Abstracts Service
INSPEC: Acquisition Section, Institution of Electrical Engineers, U.K.
Engineering Societies Library, U.S.A.