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SUMMARY REPORT
OF THE
REFRACTORY METALS SHEET ROLLING PANEL

MATERIALS
ADVISORY
BOARD

National Academy of Sciences—
National Research Council
Washington, D. C.

MATERIALS ADVISORY BOARD

DIVISION OF ENGINEERING

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SUMMARY REPORT

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REFRACTORY METALS SHEET ROLLING PANEL

Prepared By The

MATERIALS ADVISORY BOARD

Division of Engineering
National Research Council

as a service of

The National Academy of Sciences

to the

Office of Defense Research and Engineering
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This report completes a study undertaken by the Materials Advisory Board for the National Academy of Sciences in execution of work under Defense Supply Service Contract No. DA-49-083 OSA 313 between the Department of Defense and the National Academy of Sciences.

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CONTENTS

SUMMARY REPORT

REFRACTORY METALS SHEET ROLLING PANEL

Introduction

The Materials Advisory Board Refractory Metals Sheet Rolling Program was originally established at the request of the Department of the Navy, Bureau of Naval Weapons, to identify causes responsible for variation in refractory metal sheet and to develop remedies for these difficulties. It was intended, in general, to develop a comprehensive technology for making high-quality, reproducible, widely usable material, with all the implications therein, responsive to the established requirements of weapons and vehicle designers. The program subsequently was expanded through the Department of Defense to include the other Services, NASA, and AEC in a broadly based, integrated effort.

At this writing, six years later, the refractory metal sheet industry in the United States is a going business. In the last few years, there has been available a choice of strong alloys, wide and thin sheet (to 36" wide, and in narrower widths, down to foil gages) produced to close tolerances, a background of property data and formability experience, and finally, sufficient production know-how to permit reasonable deliveries and realistic quotations. Several of the currently available alloys were unknown at the start of the program. It is believed that the coordinated sheet rolling program under the guidance of the Refractory Metals Sheet Rolling Panel and its eleven Subpanels has made a significant contribution to this progress.

It is the purpose of this document to summarize the modus operandi, the accomplishments of the program, and finally, recommendations of the Panel for completion of the present program and continuation of related development activities. A more detailed summary of the main Panel and Subpanel activities and recommendations may be found in the Final Report of the Refractory Metals Sheet Rolling Panel (ref. 1), and a summary of contractor technical achievements will be found in a report prepared by the Defense Metals Information Center (ref. 2). Reference 3 summarizes the Subpanel reports which have been issued. It is hoped that this present document will entice the interested reader into a more detailed study of those references.

Need

At the inception there was a need for refractory metal sheet for certain research and development vehicles or devices such as the X-20 hypersonic glider, ramjets, and solid rocket components plus, certainly, the knowledge that with the constantly upward trend in operating temperatures, requirements would be present if quality sheet of the proper alloys could be provided.

The recently completed studies of the MAB Aerospace Applications Requirements Panel (ref. 4) outlined requirements for all materials for propulsion systems (turbojet, turborocket, turboramjet, ramjet, liquid rocket, solid rocket, and electrical propulsion) and vehicle systems intended for operational capability in 1970. They reviewed devices, components, operational and environmental regime of components, and looked at fabrication requirements. For the propulsion systems alone, they specified four sheet and plate requirements, three tubing requirements, three forging requirements, four coating requirements, and two thermionic device requirements for refractory metals. In reviewing fabrication requirements, it was found that 18 of 44 were due to the use of refractory metals. It was concluded that refractory metals will be a pacing item. The report broke down the problems of priority and identified seven items on refractory metals in priority I.

At the start of the program, very few refractory metal alloys were available, surface and dimensional control was poor, and worst of all, product quality was extremely variable. This was the era when unalloyed molybdenum was beginning to be replaced by the Mo-0.5Ti alloy, Cb-1Zr was the columbium alloy, there were no tantalum alloys, and no sizeable tungsten sheet.

A major quality problem in the limited compositions available was lack of uniformity. Variable formability and tendency to delaminate or crack during shearing and forming were persistent problems in attempted applications. These problems were most pronounced with Mo- and W-based materials. All of the refractory metals considered in this program, W, Ta, Mo, and Cb, are body-centered cubic metals and at least W, Mo, and Cb exhibit a ductile-brittle transition temperature. It is desired that this DBTT be below room temperature to facilitate handling and forming. Molybdenum sheet was found to have an extremely variable DBTT, usually above room temperature.

Surface contamination was another persistent problem. Columbium and tantalum alloys are particularly prone to contamination from oxygen and nitrogen when heated, and some molybdenum alloys are also susceptible. Such contamination reduced bend ductility and formability. The lack of uniformity also affected mechanical properties, and as a result many designers felt that refractory metals were not ready to be specified.

Method of Operation

The Panel decided that the program should be divided into three phases for each alloy:

Phase I - Development and documentation of a production practice for high-quality sheet and production of a quantity of sheet to demonstrate and establish quality and uniformity.

Phase II - Measurement of preliminary design data for the "pedigreed" sheet from Phase I.

Phase III - Establishment of limits of formability and definition of forming and joining procedures for sheet, followed by tests of fabricated structural elements. In some cases prototype aerospace vehicle or propulsion system components were to be designed, fabricated, and evaluated.

Alloy Selection

Of major importance was the decision as to which refractory metals or alloys should be fed into the program. This portion of the activity was the responsibility of the Subpanel on Alloy Requirements and Selection. This group has repeatedly surveyed the requirements for these materials by consulting the consumers and by referring to the product of the Aerospace Applications Requirements Panel of the Materials Advisory Board (ref. 4). They conducted, at the initiation of the program, a survey to learn the status of refractory metal alloy development in this country. Based upon these surveys, they decided it was desirable to set target properties for six specific classes of alloys:

1. Fabricable molybdenum
2. High-strength molybdenum
3. Fabricable high-strength columbium (originally separated into two classes)
4. Tantalum
5. Unalloyed or dilute tungsten
6. High-strength tungsten

The targets served two purposes: (1) they provided the industry with specific objectives permitting them to focus their efforts for alloy development, (2) they comprised specific tests for which data that should be obtained to permit valid comparisons to be made. The stimulus for response by the industry was the opportunity for Panel endorsement and thus for Government support for Phases I, II, and III for the selected alloys.

The targets were submitted to the industry and a date for review of a particular alloy class (e.g., high-strength fabricable columbium alloys) was announced. On the selected date all organizations offering candidate alloys were given time to present to the Subpanel the fabrication history, experience, and properties for their candidate. In closed session, the Subpanel compared the candidates and made their selections. Copies of the minutes of this meeting, including Subpanel recommendations, were sent to all participants and to the main Panel and Government contractors for action. (The documented history of these meetings and recommendations may be found in ref. 1.) By this process, a large number of candidates were narrowed down to a very few that most nearly met the targets. The selective focusing upon a very few alloys by an impartial body is considered to be a very important feature of such a development scale-up program, and especially important when the potential market is small, the need great, and the cost of the final product in 0.040-inch sheet is in the range of $30 to $280 per square foot.

Alloy selection has been an intensive process spanning several years. It required an estimation of future (and unknown) requirements, a knowledge of present capabilities, and a need to balance producibility against high properties. Those involved in the program were impressed with the manner in which industry responded to the challenge. Once clear objectives had been established, producers, whether under contract or not, made rapid progress so that within a few years several alloys in each class were available for selection. It is significant to note that at this date the target properties have been achieved for all classes except high-strength molybdenum.

Program Conduct Through the Three Phases

As will be appreciated, it is a long road from a laboratory sample to commercial availability of large sheet with good surface and flatness and close tolerances and reproducible properties. Largely, Phase I of the Refractory Metal Sheet Rolling Program was concerned with this aspect of the overall development of an alloy into a usable engineering material. During scale-up, the composition may change, mechanical properties do not always hold up, segregation is often a problem, and other, similar problems occur. Nevertheless, in this period, we have seen the process development accomplished for alloys of molybdenum, columbium, and unalloyed tungsten, with tantalum alloys not far behind.

Following completion of Phase I, several of the alloys were recommended for additional contract programming through Phases II and III. The contractors regularly documented their progress and DMIC has issued a series of reports describing contractor achievements. A report summarizing all contractor progress to date will be released by DMIC in mid-1966. This report itemizes quantity of material produced, and records processing development, properties achieved, and quality (in terms of flatness, gage control, etc.) for Phase I contracts; more complete property evaluation by Phase II contractors; and fabricability as determined by Phase II contractors.

General Program Achievements

To highlight the progress under the program, we may compare in Table 1 the current status of the alloys for high-quality sheet with their status when the program began in November 1959. This Table shows that several of the alloys which have advanced within the time period of the program to a point where sheet can be produced in large sizes with good quality and uniformity were unknown at the start of the program. The program results in terms of material quality (flatness, gage control, and reproducibility) are summarized in refs. 1 and 2.

Most important, the effort was focused. Only a few carefully chosen alloys were selected for development; only a limited number of the most important properties were measured, but in a way to permit needed comparisons, and a real effort was made to avoid unknown or unneeded duplication. Certainly the Government saved much money and time because of this selectivity and coordination. As a result there now exists a production base that can turn out a quality product. This was the prime objective of the program.

It would be difficult to say with conviction that the job is finished. The major objectives have been met but the Panel has recommended a modified Phase I and Phase II activity for several materials (see Table 2) that remain to be implemented. The Panel has recommended that specific responsibility be assigned for collection and dissemination of such production and property information beyond the formal lifetime of the Panel.

In Table 2, the Panel has not recommended further work in high-strength tungsten and molybdenum. This is because requirements were not specific enough to justify production development at this time. The AARP report, however, suggests that such material will be a firm requirement soon.

Ductile high-strength tungsten alloys containing about 5 per cent rhenium have been reviewed by the Alloy Requirements and Selection Subpanel. It was recommended that additional laboratory optimization be conducted, and that a selected alloy or alloys be scaled up at least to the pilot level for demonstration of feasibility and determination of property data.

The Panel clearly saw an immediate need for refractory metal tubing of the same alloys of columbium and tantalum endorsed in the sheet program. This coordinated activity should continue--the preferred method is to continue the Tubing Subpanel.

It is important that the alloys in sheet configuration be formable and that the formability be documented. The results of the Phase III studies that are determining the formability limits demonstrate that the alloys of the program are considerably superior to material available before the initiation of the program. (Details may be found in refs. 1 and 2.)

Auxiliary Subpanel Activities

A particularly important contribution of the Panel activity has been the output of the subpanels. During the tenure of the main Panel, eleven subpanels were created to aid in guidance, to provide standards, or to survey the state of the art and recommend needed research.

The Test Methods Subpanel has provided guidelines for testing or refractory metals where none existed before. The Coating Subpanel similarly provided needed recommendations for standard tests for coated refractory metals. The Quality Specifications Subpanel has provided targets for refractory metal sheet quality and outlined sheet sampling methods. All are being widely used. The Analysis Methods Subpanel has guided round robins for measurement of capability of analysis methods in refractory metal alloys. Several of the panels have recommended needed research that has been supported by the Services. Detailed summaries of the subpanel activities will be found in ref. 1.

Preceding discussions of contractor progress at meetings of the Panel, a DMIC representative summarized critical points.

Concluding Remarks

The Refractory Metals Sheet Rolling Panel nominally has been a coordinated effort to achieve high-quality refractory metal alloys in one product form, flat sheet. Because these same alloys are of interest for forging and tubing forms and because consolidation and ingot breakdown studied for sheet are prerequisites of all wrought forms, it can be said there has been considerable spin-off that has aided these other product forms.

The accomplishments were results of coordination among the military, the consumers, the fabricators, and the metal producers who became acquainted with each other's problems. Requirements were well publicized. The Services, NASA, and the AEC cooperated with each other to a high degree.

The general format used (originally developed for the Titanium Sheet Rolling Program) is deemed to be sound and important to success. This format consists of (1) setting targets for alloy selection based upon a consideration of requirements and potential capability; (2) selection of alloys, from all candidates offered, for scale-up development; (3) providing technical guidance for the three phases (development of production capability, design data, and evaluation of fabricability), and (4) continuous review of contracted programs to insure compliance with objectives. (Some criticisms of the details of the approach will be found in ref. 1.)

Coatings of the refractory metals are the key to successful application of refractory metals in many propulsion and vehicle systems. The

Coating Subpanel has established testing standards and evaluated specific coatings in several temperature-time spectrums. A coordinated approach in this area has been a major need for years. It is recommended that this activity continue.

Major benefits were derived by the focusing upon objectives, by narrowing the list of alloys whose development should be supported, by getting people together to define and attack common problems. In an area such as high-strength refractory metals, where the costs of the product and of development are high, the market small, and where the Government in the end is the major consumer, it seems imperative that the production industry and the consumer continue to get together in some working forum to provide mutual guidance. Some have proposed that whereas the Sheet Rolling Panel has concerned itself with bringing along process development of the required sheet, the future activity should concern itself with all product forms in refractory metals. If a "working forum" concept for future activities in refractory metals can be developed, a forum where less time-consuming concer with contractor problems will occur, more attention can be paid to selected applied materials research and process development.

The Panel has recommended that a "working forum" or a standing "Refractory Metals Requirements and Selection Panel" be created to review regularly and inform among the Government, consumer, producer, and R&D groups, in the area of refractory metal developments leading to all needed product forms. The "minutes" of such meetings should be available to all to provide maximum information for guidance of both industry in-house and Government programs. It has been proved that proper action will follow if objectives can be clarified and made known to those who must respond. It is deemed an essential feature of such activity that an "Alloy Selection Group" would impartially select specific compositions to recommend for Government support for process developments. Costs are too great and market too small to permit the luxury of process development of a large variety of compositions for a specific product form. The Panel suggests that this approach may, indeed, be appropriate for a wide variety of materials required in Government programs, particularly where there is critical need and a small market.

Recommendations

The Panel respectfully proposes the following:

1. Complete the current program, i.e., a modified Phase I and Phase II activity for several materials. For detailed comments see ref. 1.

2. Continue coordinated coating program.

3. Continue coordinated tubing program.

4. Create "working forum" or a standing "Refractory Metals Requirements and Selection Panel" (RMRSP) to review regularly and inform, among the Government, consumer, producer, and R&D groups, in the area of refractory metal developments leading to all needed product forms.

Additional recommendations will be found in the specific subpanel summaries, ref. 1.

References

1. "Final Report of the Refractory Metals Sheet Rolling Panel" MAB-212-M Materials Advisory Board, National Academy of Sciences-National Research Council, February 1966.

2. "Summary of Contractor Results in Support of the Refractory Metals Sheet Rolling Program", Defense Metals Information Center Report No. 231 to be issued in 1966.

3. "Joining of Refractory Sheet Metals" MAB-171-M, March 20, 1961
"Report of the Subpanel on Analytical Techniques, Refractory Metals Sheet Rolling Program" MAB-178-M, November 15, 1961
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"Evaluation Procedures for Screening Coated Refractory Metal Sheet" MAB-189-M, February 15, 1963
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"Recommended Quality Standards for Tungsten Sheet Produced in the Refractory Metals Sheet Rolling Program" MAB-196-M, January 22, 1964
"Procedures for Evaluating Coated Refractory Metal Sheet" MAB-201-M August 10, 1964
"Status of Refractory Alloy Tubing - 1964" MAB-208-M, August 18, 1965
"Coating Technology - 1965, Oxidation-Resistant Coatings for Refractory Metals" MAB-210-M, November 1965
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All published by the Materials Advisory Board, National Academy of Sciences-National Research Council.

4. "Design Requirements" (U) Vol. 2 of 4 (Classified) MAB-200-M(AAR-2) Report of Aerospace Applications Requirements Panel, Materials Advisory Board, National Academy of Sciences-National Research Council September 1965.

Table 1

History of Alloys Identified for Production Development by Sheet Rolling Panel

Alloy Class	Status November 1959	Status - 1965
Fabricable molybdenum		
Mo-1/2Ti	Large sheet poor quality	Completed production program (24 x 72" sheet)
TZM (Mo-0.5Ti-0.1Zr-0.03C)	Small sheet	Completed production program (24 x 72" sheet)
Tungsten		
Unalloyed	Lab. size sheet	Completed production program (18 x 48" sheet)
Fabricable & weldable columbium		
D-43 (Cb-10W-1Zr-0.1C)	Unknown	Completed production program (24" wide)
Cb-752 (Cb-10W-2.5Zr)	Unknown	Completed production program (24" wide)
FS-85 (Cb-28Ta-10W-1Zr)	Unknown	Completed pilot production (18" wide)
Tantalum		
T-222 (Ta-10W-2.5Zr-0.01C)	Unknown	Completed pilot production
GE-473 (Ta-7W-3Re)[a]	Unknown	Completed pilot production

[a] Development funded by G. E.

Table 2

Panel Recommendations in Current Program

Alloy	Phase I (Production)	Phase II (Prelim. Design Data)	Phase III (Fabrication)
Fab. Mo			
1/2%Ti	Universal Cyclops	Southern Research	McDonnell
TZM	Universal Cyclops	Southern Research	McDonnell
Unalloyed W			
P.M.	Fansteel	Southern Research	Solar
Arc Cast	Universal Cyclops	Not Recommended	Super-Temp
Tantalum			
30Cb-7½V	Wah Chang	Not Recommended	----------
T-222	Not Recommended	Recommended[a]	Not Recommended
GE-473	Not Recommended	Recommended[a]	Not Recommended
Columbium			
D-43	du Pont	Recommended[b]	Not Recommended
Cb-752	Haynes (pilot)	Recommended[b]	Not Recommended
FS-85	Recommended	Recommended[b]	Not Recommended
High Strength Mo			
Arc Cast TZC[c]	------------	------------	------------
Powder Met. TZM[c]	------------	------------	------------
Ductile W[d]	------------	------------	------------
High Strength W[d]	------------	------------	------------

Table 2 (continued)

Notes

[a] Recommended Phase I production (not necessarily process development) of sufficient quantity of material to fix and define the production process and to provide material for Phase II evaluation of this pedigreed material.

[b] Recommended collection and assembly of Phase II data (and Phase III where possible) of material already produced in Phase I.

[c] Identified as candidate for preproduction program when requirements for high-strength molybdenum warrent.

[d] Alloys were identified that have exceptional promise. After additional laboratory optimization has been completed, at least a pilot study is recommended. (See MAB-212-M, p. 120)

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SUMMARY REPORT OF THE REFRACTORY METALS SHEET ROLLING PANEL

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Summary Report of a Final Report June 1959 - December 1965

5. AUTHOR(S) (Last name, first name, initial)

Materials Advisory Board Refractory Metals Sheet Rolling Panel

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	Assistant Director (Materials) ODDR&E, The Pentagon, Washington, D. C.

13. ABSTRACT

The method of operation, accomplishments and recommendations of the Panel are outlined (summarizing the contents of MAB-212-M, "Final Report of the Refractory Metals Sheet Rolling Panel").

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Unclassified

Security Classification

14 KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
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Molybdenum alloys						
Columbium alloys						
Tantalum alloys						
Tungsten alloys						
Mechanical properties						
Chemical analysis						
Test methods						
Oxidation-resistant coatings						
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