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Report No. 8926-145

**Material - Finishes and Coatings - Oxidation
Resistant for Molybdenum**

Literature Survey

D. S. Pratt, E. Shoffner, E. E. Keller

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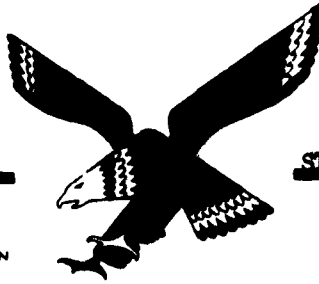
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Abstract:

Some of the advantages and disadvantages of chromium-base, molybdenum disilicide, nickel-alloy, precious metals and ceramic oxidation resistant coatings for molybdenum are discussed along with methods used for their application. The restriction of the use of the coatings discussed to applications involving only simple shapes is pointed out.

Reference: Pratt, D. S., Shoffner, E., Keller, E. E.,
"Literature Survey of Coatings That Will
Prevent Molybdenum From Oxidizing At Elevated
Temperatures," General Dynamics/Convair
Report MP 58-237, San Diego, California,
25 June 1959. (Reference attached).



STRUCTURES & MATERIALS LABORATORIES

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A DIVISION OF GENERAL DYNAMICS CORPORATION
SAN DIEGO

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**LITERATURE SURVEY OF COATINGS
THAT WILL PREVENT
MOLYBDENUM FROM OXIDIZING
AT ELEVATED TEMPERATURES**

MODEL: R.E.A. 7038

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FOREWORD

This report was written to summarize literature on coatings which prevent the oxidation of molybdenum at elevated temperatures.

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SUMMARY

Many types of high temperature protective coatings for molybdenum are available. The specific coating used will depend on the type and duration of protection desired or needed.

Chromium based coatings have a moderate resistance to oxidation, a thermal expansion factor similar to molybdenum, but tend to be brittle. Molybdenum disilicide has a good thermal coefficient of expansion match with molybdenum, and has excellent oxidation resistance. However, molybdenum disilicide is extremely brittle. Alloys with a nickel base do not fit as well with the thermal expansion of molybdenum, but do offer good bond strengths and medium oxidation resistance up to 2000°F. Protective coatings made from precious metals offer good oxidation resistance, a ductile bond, and thermal expansion fit. However, their costs are extremely high. Ceramic or glass type coatings can be custom designed for definite operating temperatures up to 2500°F. These coatings and refractory oxide coatings can be designed to give a good thermal expansion match. Hardness, brittleness or inherent high porosity are the chief drawbacks of these coatings.

The methods used to apply the coatings include electrodeposition, flame spraying, vapor deposition, cladding, enameling, and liquid phase diffusion. A duplex layer of nickel over chromium tends to overcome the porous nature of electrodeposited coatings. This coating is still weak in thermal cycling. Flame sprayed coatings are difficult to apply to hidden areas. Systems of Ni-Cr-B, Ni-Si-B, Al-Cr-Si and Mo-Si are quite successful when applied by flamespraying. Vapor deposition of siliconized and chromized coatings have shown good coverage and uniformity of thickness. Claddings are only applicable to simple shapes, but on these, clad coatings offer good protection up to the temperature limit of the cladding. Sprayed enamel, or refractory oxide coatings produce dense coatings, but ones that are brittle and difficult to keep uniform on the edges and corners. The production of a protective coating in a liquid phase has not been as practical as other methods.

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INTRODUCTION:

Molybdenum is one of the few metals having a very high melting point, its melting point being 4750°F. It has good strength properties at temperatures over 1600°F as shown in Figure 1. However, these values are only obtained under conditions that prohibit the formation of molybdenum trioxide. Molybdenum rapidly converts to the dioxide and trioxide form when heated. The oxide coating increase follows a parabolic curve and gives some self protection below 932°F. Above 932°F, the trioxide begins to volatilize. At 1415°F, the volatilization equals the rate of formation of the trioxide. Therefore, except for short periods of exposure, molybdenum is valueless in an oxidizing atmosphere over 932°F. When molybdenum is to be used over 932°F, and in an oxidizing atmosphere, protection must be applied to obtain the full capability of the material.

OBJECT:

This investigation was made with the purpose of gathering information on methods of protecting molybdenum from high temperature oxidation and to suggest possible courses of future action.

REQUIREMENTS FOR PROTECTIVE COATINGS:

Any coating considered should fall within the following general requirements:

1. Be without pinholes or porosity.
2. Have an operating life of at least 500 hours and preferably 1000 hours at 1800°F to 2000°F.
3. Be thermal shock resistant
4. Have an impact resistance against small particles.
5. Be erosion resistant against combustion gases, sands, metals and products of combustion.
6. Have 1% - 2% ductility at 1800°F.
7. Must have fatigue strength.
8. Applicable at temperatures below the recrystallization temperature of molybdenum.
9. Not interact with molybdenum in such a way as to impart brittleness.
10. Be easy to apply and handle before bonding to base metal.
11. Provide an acceptable finished surface.

REQUIREMENTS FOR PROTECTIVE COATINGS: (Cont'd)

The various laboratories working on this problem have developed methods of testing for each of these requirements. (5)

CHARACTERISTICS OF COATING SYSTEMS:

Chromium:

Coatings of chromium seem to offer the best properties and compatibility with molybdenum. The melting point of chromium and chromium-molybdenum alloys, 3100°F, is high enough for most applications. The thermal expansion fit is fair. Thermal cycling resistance is only fair because the chrome becomes embrittled during high temperature service. (10) This can lead to serious mechanical defects in the molybdenum. (8) (30)

Nickel and Nickel Based Alloys:

Unalloyed nickel has good resistance to MoO₃ vapors, and forms a molybdate which is stable up to the eutectic of the molybdenum-nickel system, 2400°F. This coating has poor thermal shock resistance. (26) Nickel-chrome alloys have better oxidation resistance than unalloyed nickel. Nickel coatings have been improved by being coated with aluminum. (9) Nickel and its alloys are ductile and stand up well to ballistic impact. The compound formed at the nickel-molybdenum interface is brittle and small amounts of nickel tend to embrittle the molybdenum itself. (11) (31)

Silicon:

The elements, silicon, boron, beryllium and aluminum, form stable, oxidation resistant coatings with molybdenum. (8) The greatest protection comes from MoSi₂ which is good up to 3100-3300°F. An impervious layer of SiO₂ forms on the surface to keep out the oxygen. This Mo Si₂ layer diffuses to Mo₃Si₂ and then to Mo₂Si which is not oxidation resistant. This diffusion time sets the service life of the coated part. There is a good thermal expansion match between the MoSi₂ and molybdenum and this contributes to good thermal shock resistance. Below 1500°F the coating is brittle, but at 2700°F, is fairly ductile and will exhibit some self healing as the molten silica bridges defects. (8)

Precious Metals:

Little time has been spent on the development of protective coatings from precious metals. Iridium, rhodium and platinum have a coefficient of expansion that is very favorable. They are extremely oxidation resistant and are ductile. The meager work with these coatings seems to indicate that a service life of several thousand hours at 2200°F might be possible. (29) (37)

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CHARACTERISTICS OF COATING SYSTEMS: (Continued)**Glass Coatings:**

Coatings of glass are oxidation resistant and are self healing at high temperature. However, brittleness and poor thermal shock resistance are major drawbacks to glass coatings. This type of coating can be made to have a close match in thermal expansion. Attention must be paid to the melting temperature of the glass or difficulty will be encountered. Too low a melting temperature will cause the glass to flow away leaving bare molybdenum while too high a melting temperature will prevent the glass from self healing small defects. (27) (28)

Refractory Oxides:

Two major advantages are present when refractory oxides are considered. They exhibit good resistance to erosion or abrasion, and have insulation properties greater than metals. In thin layers, coatings of ZrO_2 , BeO , and Al_2O_3 have good thermal shock adherence but in thick layers are subject to spalling and poor adherence. Like glass coatings they are brittle and have poor thermal shock resistance. Their inherent porosity is sometimes controlled by the addition of a glassy phase. This of course has an adverse effect upon some of the other properties.

METHODS OF APPLYING PROTECTIVE COATINGS AND THEIR CHARACTERISTICS:

There are many methods of applying these protective coatings to the molybdenum and each has its own advantages and disadvantages. Some of the coating materials have been applied by several methods. The method used depends on the degree of protection needed, shape of the part, and service life expected. The adherence, uniformity and continuity are somewhat dependent upon the skill used in applying the chosen method. The methods most commonly used are electro-deposition, flame spraying or metallizing, vapor and/or pack deposition, enameling, cladding (roll or pressure bonding), and liquid phase deposition.

Electrodeposited Coatings:

Coatings applied by electro-plating have advantages and disadvantages that are shown by the following listing. (18)

Advantages

1. Low application temperature
2. Controlled thickness
3. Coat complex parts
4. Build up of multi-layer coats
5. Produces smooth finish

Disadvantages

1. Complex procedures
2. Porous coatings
3. Difficult to coat electrical contact areas

METHODS OF APPLYING PROTECTIVE COATINGS AND THEIR CHARACTERISTICS: (Cont'd)

Aluminum over a chromium-nickel coating will reduce the rate of oxidation. However, due to difficulties in applying a satisfactory base coat of nickel this work is limited. (9)

Chromium has been widely investigated as an electroplated coating. The adherence of this coating has been good but oxidation resistance has not been reliable from laboratory to laboratory. This coating has been used as an undercoating for nickel coatings. (15) (10) (31) (12)

Nickel is difficult to electroplate in a non-porous, adherent coating. Its oxidation resistance is not as good as chromium. The best use of nickel seems to be when used over chromium. (25)

The area most widely investigated in electroplated coatings has been a dualayer system of chromium and nickel. The first coat is chromium, the second nickel. Chromium fits fairly well to both the molybdenum and the nickel, while the nickel protects and seals the chromium. This combination of coatings has good adherence, ductility, impact and erosion resistance. A weak point of the combination is the poor thermal shock resistance. This type of service results in failure at the nickel - chromium interface. (11) (32).

Flamespray Coatings:

Coatings of molybdenum by this process has been investigated by a number of laboratories. Materials used in powder, rod, or wire form, have been Ni - Cr - B, Ni - Si - B, Al - Cr - Si, Al - Si, Al_2O_3 , and Mo Si_2 . Applied in this manner the coatings are porous and rough, but homogenous. A proper diffusion treatment and finish grinding will produce a pore free, well bonded coating. Limitations on this method are the inability to coat small interiors, narrow openings, and reverse recesses. (4)

Nickel-chrome-boron, and nickel-silicon-boron are useful up to about 2000°F. The initial impact resistance, at high temperatures, is good. The thermal shock resistance is fair as is the erosion resistance. (4) (23)

In the absence of mechanical shock and erosion a flamesprayed coating of Al-Cr-Si offers protection up to 2000°F. The adherence is good and the coating exhibits a tendency to be self heating. (11)

Flamespray applied refractory oxides, while being hard and oxidation resistant, are generally porous and have poor adherence. (11)

Molybdenum disilicide when properly applied by flame spraying offers a good coating. The oxidation resistance is good. The Linde (LM5) coating seems to have overcome the poor ductility problems for it withstands straining up to the breaking of the molybdenum at 1800°F.

METHODS OF APPLYING PROTECTIVE COATINGS AND THEIR CHARACTERISTICS: (Cont'd)

Vapor and/or Pack Deposited Coatings:

Coatings of chromium and silicon can be deposited on molybdenum from gaseous compounds. One method is to decompose a prepared vapor in a hot chamber in which an active atmosphere flows freely. Another system is to pack the part in a powder and at an elevated temperature, pass through a suitable gas. (21) Coatings from either system have good adherence, are uniform in thickness and will reach areas that are difficult to coat by any other means.

Molybdenum disilicide coatings are generally 1 to 3 mils thick. The coating has excellent oxidation resistance. The major drawback to $MoSi_2$ is the extreme brittleness of the coating. Any deformation of the molybdenum will fracture the coating. (3) (14)

The pack deposition method is used for chromium coating. Initial work in this system showed the coating to be limited to 1500°F, but the latest developments have increased the temperature range. The Chromalloy Corporation has developed a compound called W-2 which, when applied by the packing system, gives satisfactory service at 2000°F. The W-2 coating is hard and brittle, but will withstand some room temperature deformation, and a great deal at 2000°F.

Clad Coatings:

A coating with good finish, and ductility can be obtained by cladding molybdenum with an oxidation resistant material, such as, platinum or nickel based alloys. The molybdenum is enclosed in the cladding material and then squeezed at 2000°F under sufficient pressure to bond the two systems together. The principal limitation to this type of protective coating is that it is limited to simple shapes and can not be cut or sheared without exposing unprotected molybdenum. Other limitations are imposed by minimum melting temperatures, bond strengths and the brittleness of the interfacial diffusion layers. (7)

Platinum offers a good thermal expansion match to molybdenum, has ductility and is oxidation resistant. Little is known about the diffusion layer which would be established between the molybdenum and platinum. Also the rate of diffusion of oxygen through the platinum may cause interboundary oxidation of the molybdenum. The expense factor has been a major hold up to extensive laboratory investigation of platinum as a protective coating for molybdenum. (29)

Nickel and nickel based alloys do not match molybdenum in thermal expansion. The interfacial bond produced by cladding offers fair thermal shock resistance before failure occurs. The service life of a well applied clad part is pretty well determined by the thickness of the cladding. The interpositioning of a diffusion layer retards the growth of interfacial molybdenum - nickel compounds and further extends the life of the part. (26)

By undercutting the edge of molybdenum then filling with an oxidation resistant weld material, protection is provided to clad molybdenum that has been cut or sheared. (7)

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METHODS OF APPLYING PROTECTIVE COATINGS AND THEIR CHARACTERISTICS: (Cont'd)

Enamel Coatings:

Enamel coatings are sprayed on and then fired at an elevated temperature to provide a homogenous, pore free, glassy type of cover to the molybdenum. Being glass means they have the limitations of glass - no ductility at low temperatures, but they are oxidation and erosion resistant. The coatings are thicker than most other types and are limited to shapes with moderate changes in contour.

The addition of a refractory oxide improves the service life of a glass coating. A tri-layer system of glass, a refractory containing glass, and glass has given 3/4 hour service at 3450°F. (27)(28) The proper formulation of glass will provide the viscosity that will be self healing and erosion resistant at the temperatures being used.

A two layer system of enamel over chromium is also serviceable under certain conditions. A layer of glass diffused over a glass free layer of chromium gives a coating superior to either coating alone. (28)

Liquid Phase Diffused Coating:

By immersing the molybdenum part in a molten bath of the coating material, a protective coat with good bonding is obtained. The coating thickness is generally dependent on the time of immersion. Among the problems of this method are the limits set by the contour of the part and the skill required to obtain a uniform coating. It does offer a good method to coat interiors and concealed areas.

Aluminum-silicon alloys have been applied to molybdenum by dipping. The protection obtained has been unreliable, and is thought to be due to gross inclusions. (13)(5)

Chromium coatings have been applied by a modification of the Pack Coating system. The part is packed in chromium powder and heated to about 3100°F. A liquid chrome-molybdenum phase envelopes the part in a tightly adhering, uniform coating. This coating is dense and rich in chromium. It is subject to the same drawbacks as other chromium coatings, fair oxidation resistance, but with oxide spalling in thermal cycling and embrittlement after a period of service. (21) (34) (8)

CONCLUSIONS

A number of coatings for the prevention of the oxidation of molybdenum at elevated temperature are now available. These coatings can be applied in a number of different ways. Table 1 from the report by Bartlett, Ogden, and Jaffee (2) has listed the coatings, methods of application, and possible service life. Each of the available coatings has a drawback of some type. The type of coating and method of application selected will be determined by the service life required and the part design. Table 1 will serve as an aid in making a selection, but the service life listed must be viewed with caution. Not enough testing has been done under standard controlled conditions to firmly establish the service life of each coating.

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CONCLUSIONS: (Cont'd)

Before using any coating it should be tested under simulated service conditions in order to establish the service life and to evaluate the method of application selected.

RECOMMENDATIONS:

There are two areas in which further work can be considered.

1. Aid to Industry:

The WADC Materials Laboratory has established a committee to study all phases of the conditions important to the service of protected molybdenum at elevated temperatures. The conditions of possible service of nose cones, leading edges, radomes, exhaust nozzles, and so on, will be studied. The committee is to establish standards for all areas that will affect the service life of coated parts. Having standards for testing will greatly aid industry in selecting coatings for various types of service. (2)

Convair has the type of high temperature testing devices, i.e., plasma jet and rocket motors, which will be used by this WADC committee in their evaluation program. If the WADC Materials Laboratory is granting contracts for a standardization study on protective coatings for molybdenum, it would seem to be to Convair's best interest to try to become a part of the program. Being part of the program would keep Convair abreast with the latest "state of the art", and provide an excellent training for personnel before entering into any private investigation of protective coatings.

2. Development of Coatings:

Coatings from the precious metals is an area least investigated. The cost might be considered high at first glance, but when compared to the strength and weight savings that can be obtained from the use of molybdenum, the cost becomes a smaller factor. The future development of nose cones, high speed leading edges, ramjet motors, deflectors, domes, etc., will require materials that will have strength at elevated temperatures. Molybdenum has the greatest potential of known materials for structural application on basis of mechanical and physical properties, in the temperature range from 1800°F to about 2500°F.

The development of modifications in the chrome frit coatings is a possibility that should be investigated. The addition of refractory materials would give longer life at 1800°F and higher temperatures. This coating could be combined with other systems as a second coat. A metalized coating with lower continuous oxidation life could be helped by the chrome frit covering. The metalized coating in turn would improve the hot ductility of the chrome frit coating.

Fig. 1 Stress Rupture strength of molybdenum and three molybdenum-base alloys as compared to the 13 super-strength alloys included in ASTM Special Technical Publication No. 160 "Molybdenum and molybdenum-base alloys produced by arc-casting process and tested after mechanical working and stress relieving." (16)

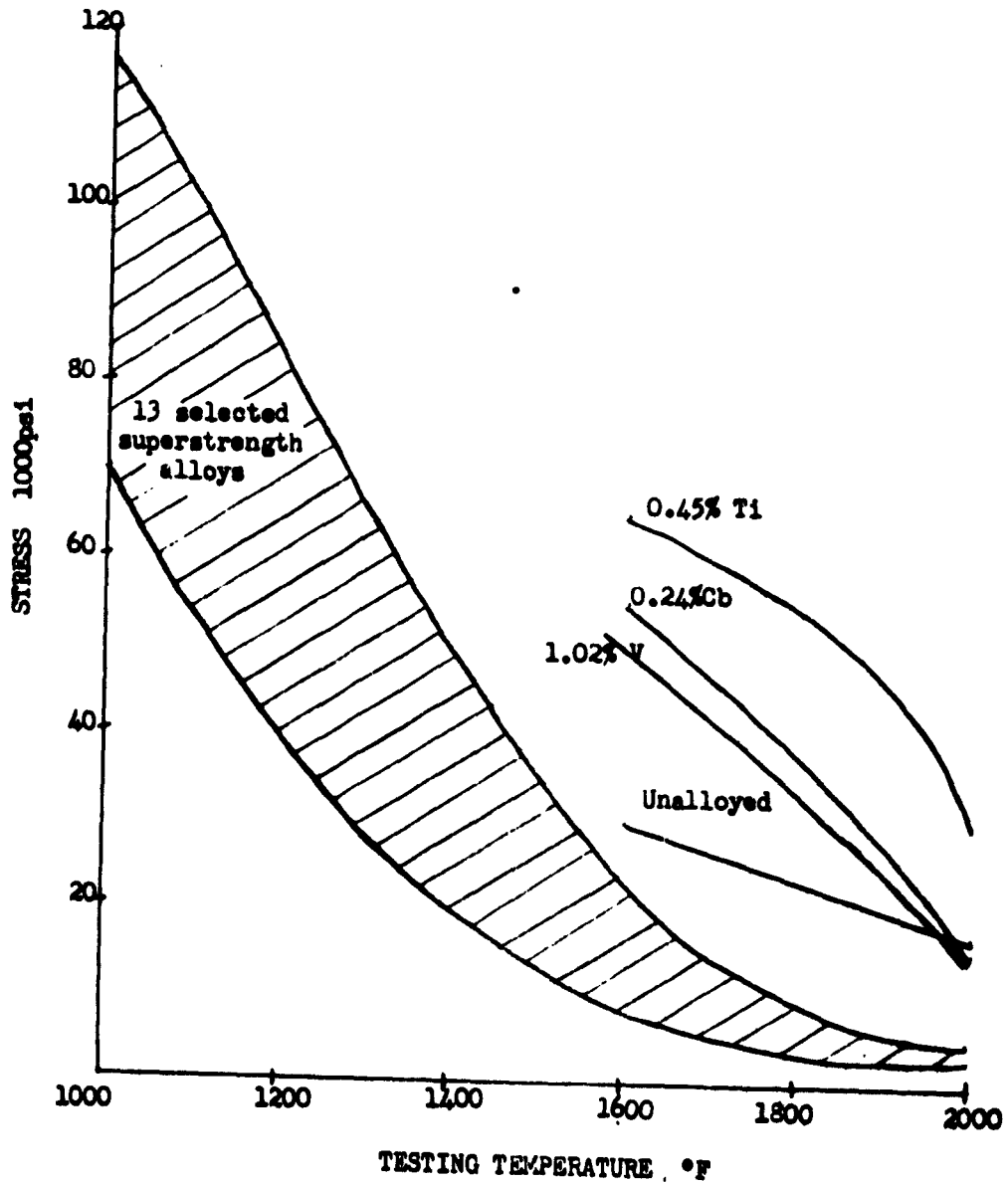


Fig. 2
OXIDATION RATE ON UNPROTECTED
MOLYBDENUM SHEET IN STILL AIR
AT 1300°F and 1500°F (19)

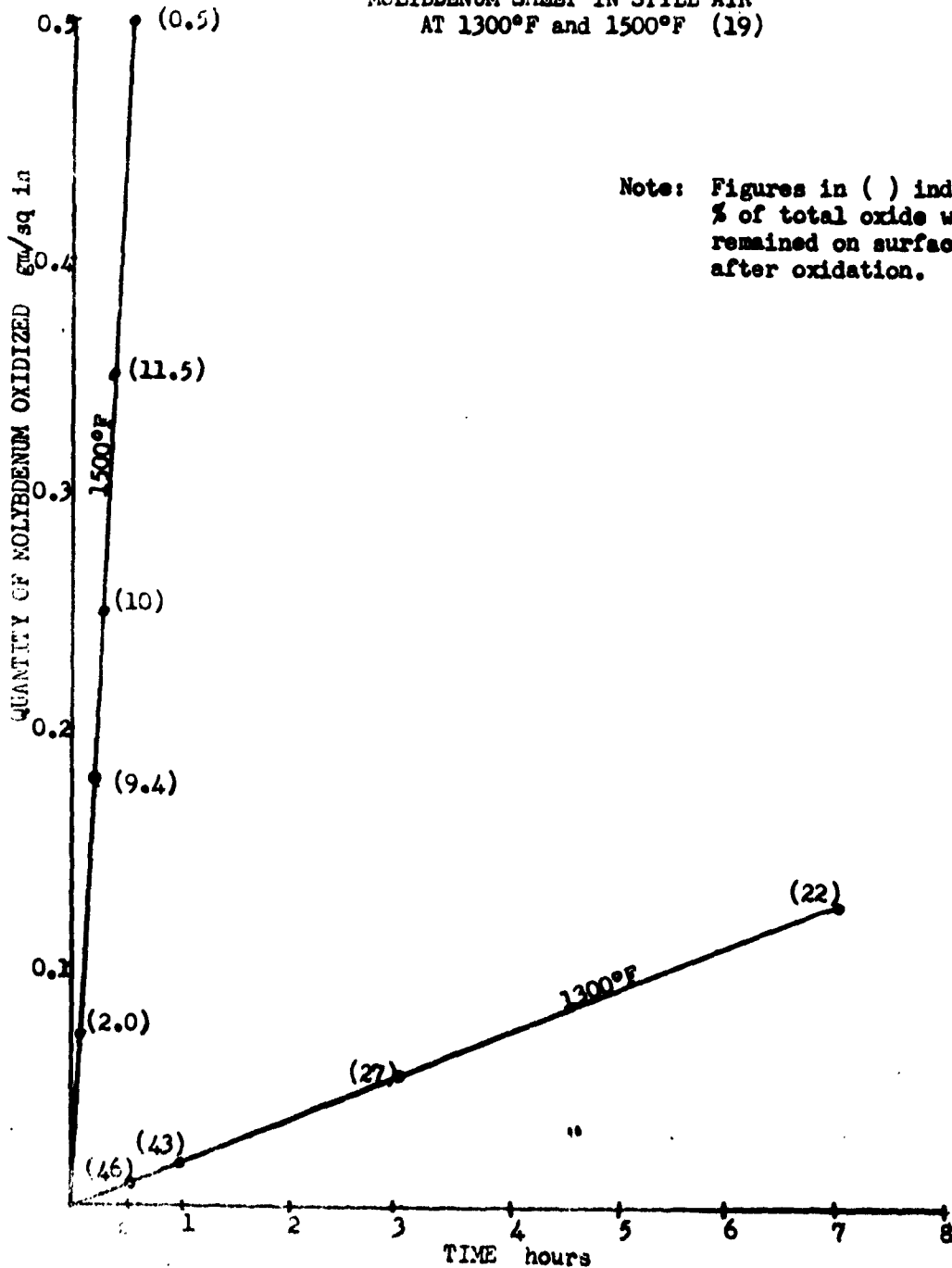


Table 1

COMPARISON OF COATINGS ON MOLYBDENUM

Coating	Continuous Oxidation Life(a), hours							
	1600°F	1800°F	2000°F	2200°F	2400°F	2600°F	2800°F	3000°F
Electroplated Cr-Ni (1mil Cr, 7 mil Ni)		1000	350	100	>1			
Metallized Al-Cr-Si (5-10 mils)		>500	>500	~100	350	>4	4(b)	4(b)
Metallized Ni-Cr-B (5-10 mils)		>500	>500	<25	<1			
Metallized Ni-Si-B (5-10 mils)		>500	1000	<10	<1			
Metallized LM-5 (5mils)					500			
Vapor deposited MoSi ₂ (1-3 mils)	6500		3700		2000			100
Vapor deposited Chromalloy W-2 (1-3 mils)			7200		500			> †
Glad Ni base (3 mils)		>2000	250					
ZrO ₂ frit (10 mils)	70				> †	> †	> †	> †
Cr frit (10 mils)	~200	~4000			~5	~5	~5	~1

(a) May include occasional cycling for examination

(b) After extensive prediffusion

~ Approximate time

Note: Oxidation life is considered terminated when molybdenum trioxide fumes are evolved.

Table 1 cont'd
 COMPARISON OF COATINGS ON MOLYBDENUM

Coating	Probable Reliability Rating (d)	Thermal Shock Resistance		Hot Ductility, or Resistance to Failure During	
		Cycles (e)	Probable Rating	Balistic Impact (g)	Stress Induced Deformation (g)
Electroplated Cr-Ni (1 mil Cr, 7mil Ni)	Fair	~140	Fair	Good	Fair
Metallized Al-Cr-Si (5-10 mils)	Good	~500	Good	Poor	Fair
Metallized Ni-Cr-B (5-10mils)	Good	~200	Fair	Good	Excellent
Metallized Ni-Si-B (5-10 mils)	Good	~100	Fair	Good	Excellent
Metallized LM-5 (5 mils)	Good(?)	Several(f)	Fair(?)	-	Excellent
Vapor deposited MoSi ₂ (1-3 mils)	Excellent	>100	Good	-	Fair
Vapor deposited Chromalloy W-2 (1-3 mils)	Good	>30	Good	-	Good
Clad Ni-base (3 mils)	Good	~100	Fair	Good	Good
ZrO ₂ frit (10 mils)	Fair	>10	Good	-	-
Cr frit (10 mils)	Fair	>60	Good	-	Fair

- (d) Within part design limitations for process
 (e) From 80°F to 400°F and 1600°F to 3000°F ranges, usually less than 1 minute for heating or cooling
 (f) After diffusion treatment
 (g) At ambient temperature

Table 1 cont'd

COMPARISON OF COATINGS ON MOLYBDENUM

	Temperature for Self Healing °F	Hot Erosion Resistance		Facility of Adequately Protecting Corners, Recesses, Etc.	Retention of Strength of Mo alloys During Application
		Minutes in Al ₂ O ₃ Blast	Probable Rating		
Electroplated Cr-Ni (1 mil Cr, 7 mils Ni)	-	95	Good	Fair	Good
Metallized Al-Cr-Si (5-10 mils)	>2600	25	Fair	Poor	Good
Metallized Ni-Cr-B (5-10 mils)	-	60	Fair	Poor	Good
Metallized Ni-Si-B (5-10 mils)	-	50	Fair	Poor	Good
Metallized LM-5 (5 mils)	2000	-	Good(?)	Poor	Good
Vapor deposited MoSi ₂ (1-3 mils)	>2800	-	Good	Good	Poor
Vapor deposited Chromalloy W-2 (1-3 mils)	(some)	-	Good	Good	Fair
Clad Ni base (3 mils)	-	70	Fair	Poor	Good
ZrO ₂ frit (10 mils)	1800(?)	-	Good(?)	Fair	Fair
Cr frit (10 mils)	1800	-	Good(?)	Fair	Poor

Source: Bartlet, E. S., Ogden, H.R., Jaffee, R.I., Defense Metals Information Center Report No. 109, Mar 6, 59, DMIC, Battelle Mem. Inst., Columbus, 1, Ohio.

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ABSTRACT: Some of the advantages and disadvantages of chromium-base, molybdenum disilicide, nickel-alloy, precious metals and ceramic oxidation resistant coatings for molybdenum are discussed along with methods used for their application. The restriction of the use of the coatings discussed to applications involving only simple shapes is pointed out.

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