

NASA Contractor Report 182304

Fabrication of Pd-Cr Wire

{NASA-CR-182304} FABRICATION OF Pd-Cr WIRE
Final Report (Battelle Columbus Labs.)
27 p C S C L 1 1 F

N89-26078

Unclas
G3/26 0219623

Sidney Diamond and Dennen M. Leach
Battelle
Columbus, Ohio

July 1989

Prepared for
Lewis Research Center
Under Purchase Order C-02999-J

NASA
National Aeronautics and
Space Administration

FINAL REPORT
on
FABRICATION OF Pd-Cr WIRE
to
NASA LEWIS RESEARCH CENTER
CLEVELAND, OHIO
from
BATTELLE
Columbus Division
July 17, 1987

INTRODUCTION

Discussions among the staff of NASA Lewis Research Center, Battelle personnel, and Dr. M. M. Lemcoe indicated that NASA LeRC was interested in having a Pd-Cr alloy fabricated into fine wire. Battelle's experience in casting, metal working, and wire drawing were considered to be applicable to NASA LeRC's goal and Battelle submitted a letter proposal to melt, cast, and draw a specified alloy of palladium and chromium to wire of about 0.02-inch-diameter and to determine the drawing conditions for the material.

It was understood by BCD that, if the condition of the material were to be judged by NASA LeRC to be acceptable at this point, a follow-on effort could be authorized at BCD to attempt to draw the Pd-Cr alloy to very small diameter wire (0.003 and 0.001 inch). Both efforts were authorized by NASA and completed by BCD, with some modification in the final wire diameter.

The wire produced by BCD in the two (related) programs has previously been sent to NASA Lewis. This report provides a description of the processing conditions and characterization of the material at various stages of the work.

Experimental Procedure and Results

Melting and Casting. NASA LeRC provided 115 grams of Pd and 22 grams of Cr to BCD as feedstock for the desired alloy of composition 87%Pd-13%Cr (by weight). The alloy was melted and cast in an inert atmosphere in a split, tapered copper mold. On examination, the surface of the ingot was considered acceptable for processing. The ingot was then solution-annealed at 1100 C in vacuum for 8 hours and top and bottom cuts were made to provide metallurgical and chemical samples.

Chemical analysis was performed by National Spectrographic Laboratories of Cleveland, Ohio. The chemical composition of the alloy was reported to be 86.1%Pd, 13.19%Cr, 0.37%Pt, and 0.21%Fe. All values are in weight percent.

Swaging. The ingot was then swaged from 0.670 inch to 0.287 inch with intermediate anneals and the resulting bar was sanded to remove all surface blemishes. The bar was then annealed in vacuum for 3 hours at 1100 C.

The rod was alternately swaged and annealed until a diameter of 0.219 inch was reached. At this point small cracks were observed on the surface of the bar but they appeared to penetrate into the bar only 0.002 to 0.003 inch. However, in order to draw the material it was essential that surface flaws be removed so that fully sound material would ultimately be produced in the final small diameter wire product.

Therefore, the bar was centerless ground to remove 0.007 inch from the radius. Microscopic examination of the surface confirmed that the cracked volume had been removed and the new surface was again sound.

A metallographic sample of the alloy was prepared after each cold working operation and after each annealing cycle. Representative micrographs of the alloy bar and wire are presented in Appendix A at selected stages throughout the wire fabrication.

Wire Drawing. Below the diameter of 0.205 inch the only forming operation was wire drawing. In general, five passes through

successively smaller dies were made to achieve a total reduction in area between 45-50 percent. Throughout the program this was considered one drawing stage. At this point a small transverse sample was cut from the wire and annealed at 1100 C/1 hr in vacuum. It was found that this treatment resulted in the formation of ten to twenty grains across the transverse interface of the wire. Since this is generally considered to be acceptable for wire product, the active material inventory was not annealed until it was established that this condition had been achieved in a test coupon.

This upper limit of cold work was arrived at through observation of the behavior of the wire during drawing, the amount of heat developed in the wire during cold working, and the stiffness of the wire after the fifth draw. Albeit that these are all subjective and non-quantitative observations, it is through the expertise of the fabricator that they became processing tools and were applied successfully.

A take-up spool was fashioned from stainless steel bar stock and alumina inserts to hold the increasing lengths of wire during the numerous anneals which were anticipated during the course of the fabrication of the alloy. The spider spool is shown in Figure 1.

This procedure was followed until the wire diameter had been reduced to 0.020 inch. After the wire had been annealed at 1100 C/1 hr in vacuum, several ten- and fifteen-inch-long sections were cut from the inventory. These were used for measurement of the resistance of the wire and for evaluation by Dr. M. M. Lemcoe, who served as technical consultant on this program based on his considerable experience in wire technology (see Appendix B). Dr. Lemcoe's evaluation of the wire is presented in Appendix C.

Based upon the formability of the Pd-Cr alloy as evidenced by the ease with which it had been drawn and the favorable interim evaluation of the 0.020-inch diameter wire, a follow-on program was authorized by NASA LeRC.

ORIGINAL PAGE
BLACK AND WHITE PHOTOGRAPH

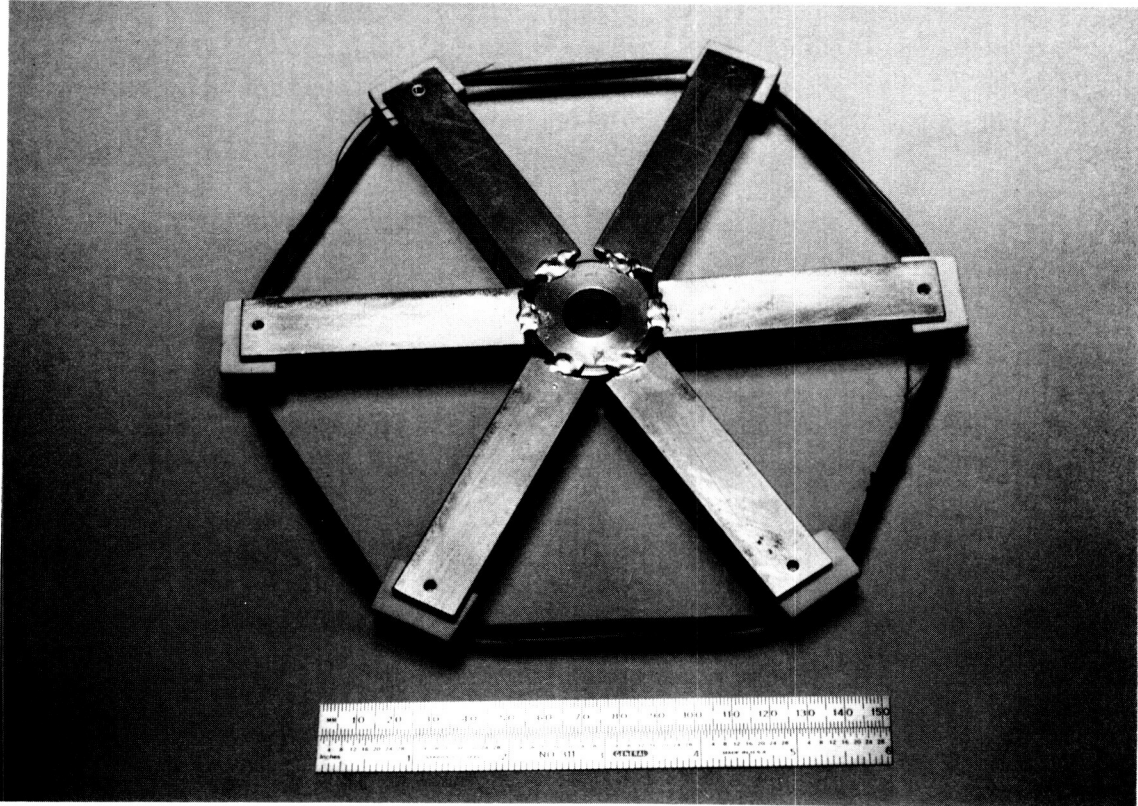


FIGURE 1. STAINLESS STEEL SPIDER SPOOL USED FOR
COILING AND ANNEALING Pd-Cr WIRE

Follow-On Program

The objective of the follow-on program was to continue the drawing and evaluation of the Pd-13%Cr alloy on a best effort basis to reach two target wire diameters, 0.003 inch and 0.001 inch. The latter target diameter was revised to 0.00176 inch by NASA LeRC as the wire fabrication was nearing completion.

Experimental Procedure and Results

The same wire drawing procedure was employed in the follow-on program as in the preceding effort which was described above. However, the annealing parameters were changed as the cross section of the wire was reduced. For instance, at 0.015-inch diameter it was determined that 950 C/1 hour in vacuum produced an acceptable recrystallized grain size. At 0.011-inch diameter 900 C/1 hour was found to be adequate. From 0.0044-inch diameter and lower, the annealing temperature was 800 C/1 hour in vacuum.

The progressive decrease in annealing temperature was determined by two considerations. The first was to establish the appropriate microstructure in the wire as a result of recovery and recrystallization following the cold work in the drawing passes. The second consideration was to minimize the potential loss of Cr which could occur during prolonged annealing at high temperature, particularly in the small diameter wires which were being produced.

After each drawing stage of 45-50 percent total reduction in area, a length of annealed wire was taken from the active inventory and set aside as an archive sample. In general, sufficient material was withheld such that the desired quantity of wire at the target diameters could be produced from the archive sample were wire in the active inventory to be compromised in the subsequent set of draws and annealing cycle.

The drawing and annealing proceeded very smoothly and without incident until the wire size of 0.003 inch was reached. At this point incipient sintering of the wire strands occurred at pressure points on

the alumina inserts of the stainless steel spider spool which was used to hold the wire during annealing. At each location that a wire strand passed over the edge of an alumina spacer, the strands bonded to one another. The pressure was greatest on the innermost strands and the sintering was most prevalent in the lower-lying regions of the spool of wire. Although the wire could be separated into its individual strands, BCD researchers considered the integrity of the wire to have been compromised. The contact points between the strands could be the source of kinking and breaking during drawing or fracture during subsequent application even if the wire were to be drawable. However, it was determined from discussions with the program monitor at NASA that the wire between the sinter points, which was about 2-1/2 - 3 inches in length, could be cut and utilized as lead wires for NASA's application.

Further, the outermost strands of the wire on the spool were unsintered and intact and sufficient to provide a considerable length of smaller diameter wire, although not the target length which had been desired. Discussions between the NASA program monitor and the BCD program manager determined that a 50-100-foot length of smaller diameter would be adequate for NASA's immediate requirements. BCD then modified the alumina inserts on the spider spool to reduce the tension on the wire during annealing and thus reduce the tendency to sinter.

At this point 185 ft of 0.003-inch diameter wire was retained to be supplied to NASA LeRC for lead wire material. Approximately 53.5 ft of 0.003-inch diameter wire was drawn to 0.0176-inch diameter, at which point the NASA project monitor decided that further reduction of the wire was not required for NASA's intended near-term applications. The inventory of 107 ft of 0.0176-inch diameter wire was then annealed in the standard manner. Three 12-inch long pieces were cut from the wire and again supplied to Dr. Lemcoe for his evaluation, which is presented in Appendix D.

The entire inventory of wire and other alloy pieces as shown in Appendix E was then shipped to NASA LeRC.

In discussing the condition of the Pd-Cr wire with Dr. Lemcoe, he stated that the resistance of the wire was somewhat lower, by about 15-20%, than comparable wire of other alloys used for strain gages. BCD

suggested that this conditions could arise from two sources: (1) the intrinsic resistivity of the Pd-13%Cr alloy and (2) the possible loss of Cr from the alloy wire during the numerous anneals at relatively high temperature required throughout the fabrication of the wire. Although the second mechanism was viewed as remote, it had to be considered a possibility until proven otherwise.

To determine if Cr loss had occurred, BCD requested that supplemental funding be allocated to perform chemical analyses on selected samples. NASA LeRC agreed and two wire samples previously prepared for optical microscopy, one of 0.144-inch diameter and one of 0.003-inch diameter, were analyzed by wavelength dispersion spectroscopy. Analysis showed the composition to be 86.69%Pd, 13.31%Cr by weight in the larger wire sample and 86.99%Pd, 13.01%Cr in the smaller diameter wire sample. These values compare favorably with the composition determined earlier on the ingot and indicate strongly that little or no Cr loss had occurred during the annealing of the wire in the course of fabrication. This suggests that the selected composition of the alloy is such as to confer a lower intrinsic resistance than encountered in other alloys for strain gage applications.

Recommendations for Future Development of Pd-Cr Alloys for Strain Gage Applications

The electrical resistance of solid solution alloys is a strong function of the amount of solute in solution in the host metal. Since the present alloy apparently possesses a lower resistance than is desirable for high temperature strain gage applications, the only way to increase the resistance of the material is to increase its solute content without causing the occurrence of any phase changes in the system. From the phase diagram presented in Figure 2, it appears possible to raise the Cr level to as high as 19wt.% from the present level of 13%. This provides a range of composition which can be explored for developing an alloy with the appropriate resistance level.

BCD would not anticipate a dramatic change in fabricability of a higher Cr alloy based on the relative ease with which the current alloy

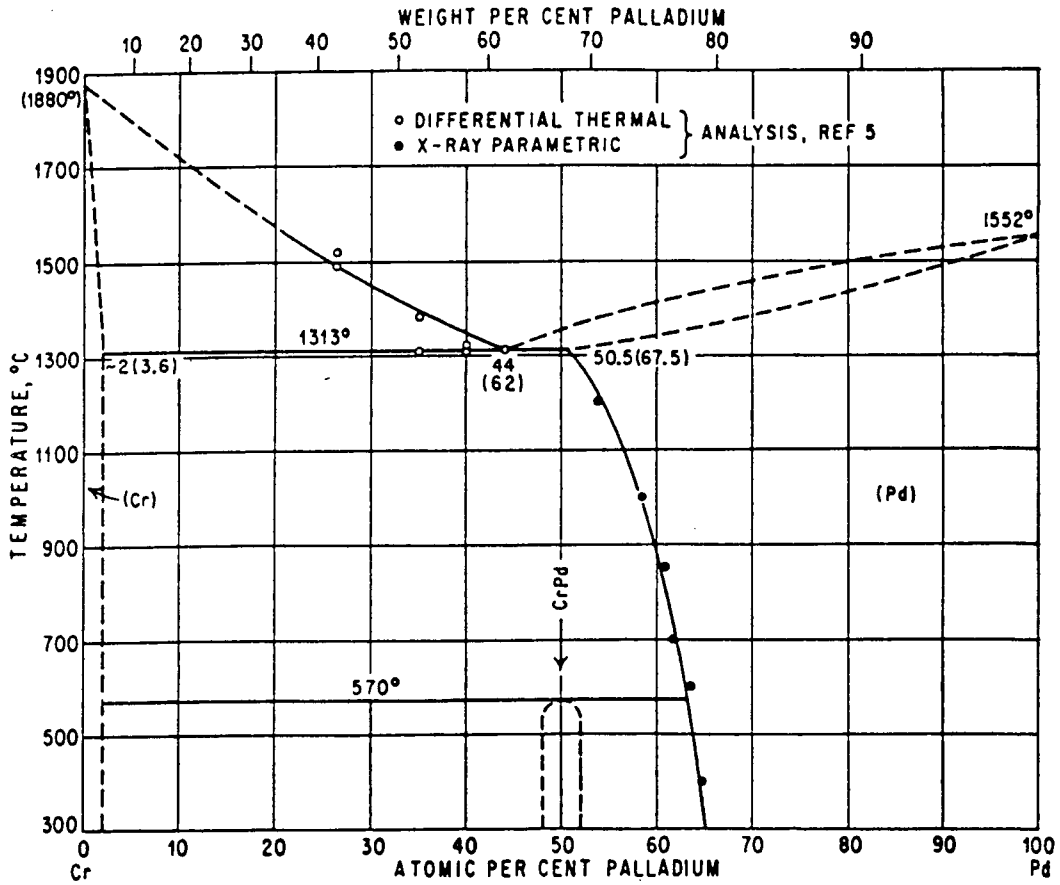


FIGURE 2. PALLADIUM CHROMIUM PHASE DIAGRAM
(From "Constitution of Binary Alloys", by Max Hansen, 1958.)

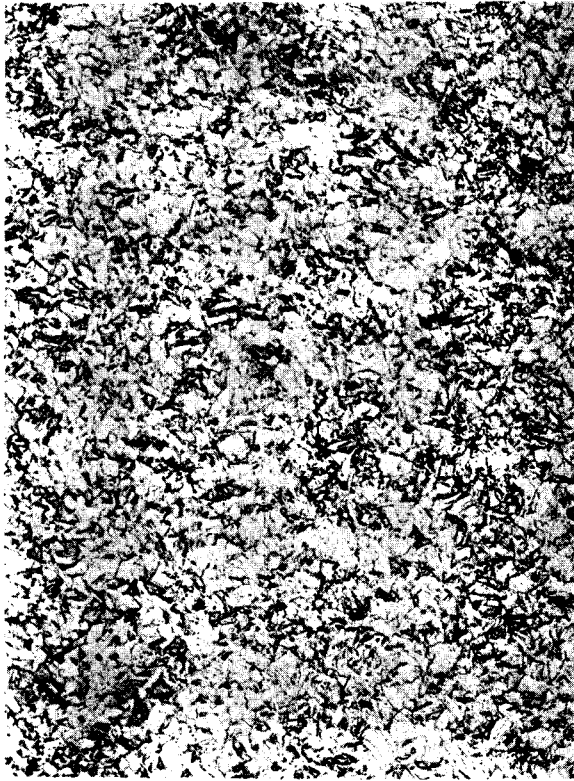
was fabricated. The major complication which could occur would be an ordering mechanism as the phase boundary is approached by alloys of increased Cr content. Ordering as a precursor to the formation of an intermetallic phase is often encountered in binary alloy systems and can strongly influence mechanical and physical properties, mostly adversely. However, no indication of ordering has been encountered in the current system. BCD considers it feasible to increase the Cr content of the experimental Pd based alloys in a controlled manner to achieve the desired electrical resistance of the potential strain gage wire. BCD recommends that the same experimental procedure employed in the completed project described above be utilized in further alloy development programs on this alloy system within the single phase solid solution region of the phase diagram.

APPENDIX A

REPRESENTATIVE MICROGRAPHS OF Pd-Cr ALLOY AT SELECTED
STAGES DURING WIRE FABRICATION

AS DRAWN

ANNEALED



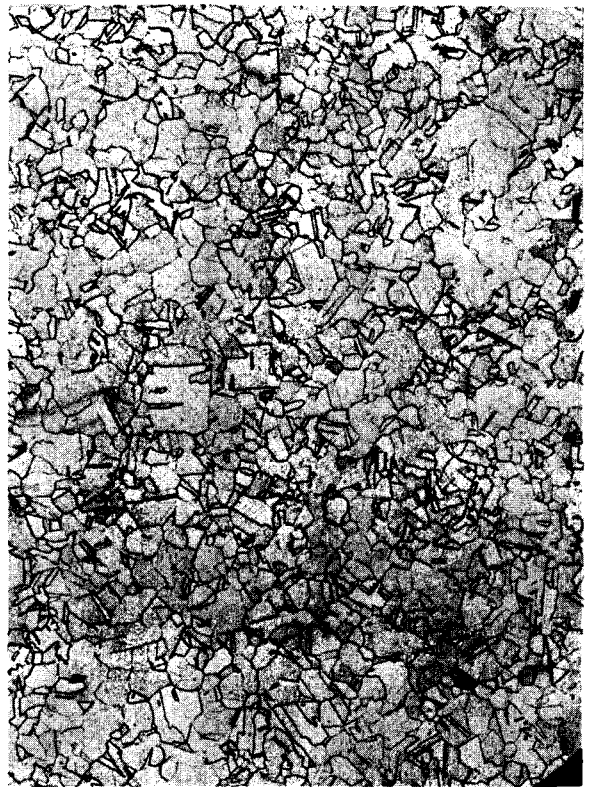
100X 0.072" Dia. 7M788



100X 0.072" Dia. 7M790



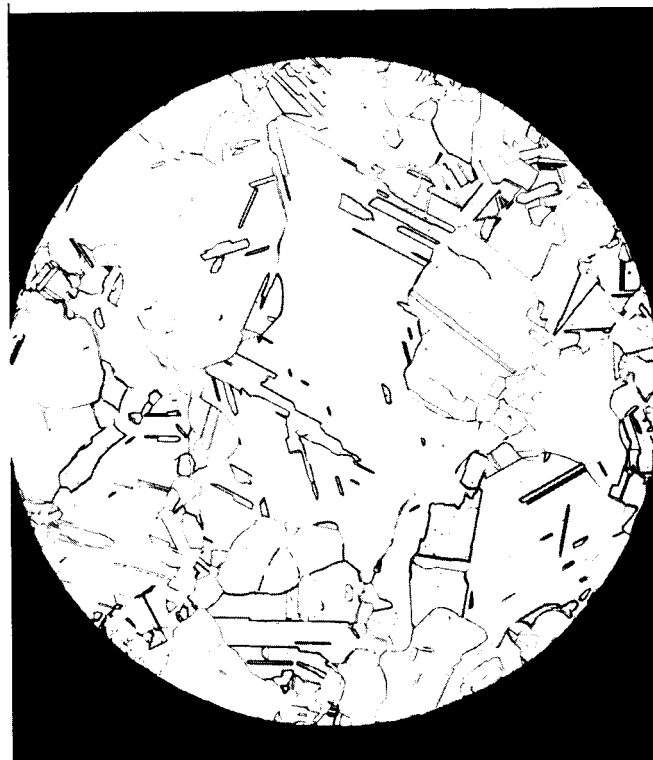
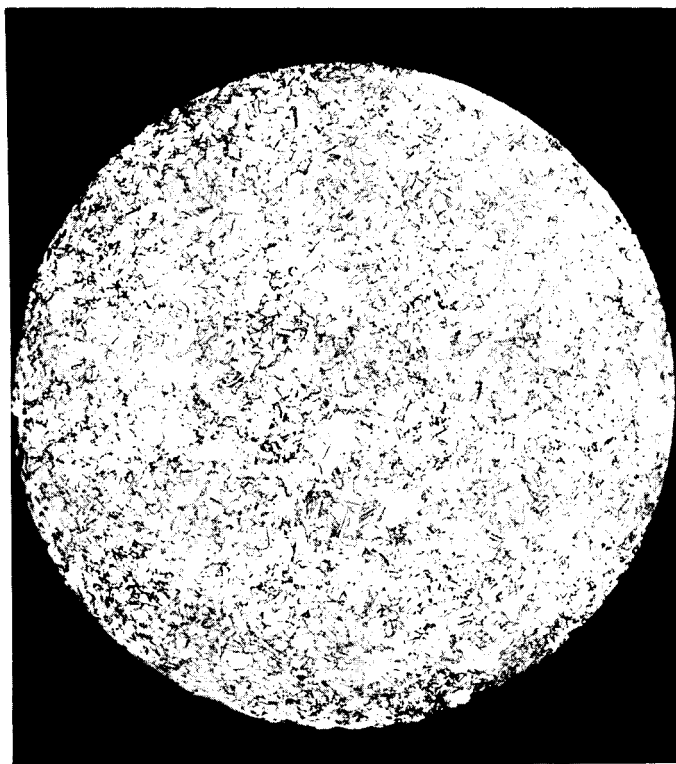
100X 0.051" Dia. 7M792



100X 0.051" Dia. 7M794

AS DRAWN

ANNEALED



100X

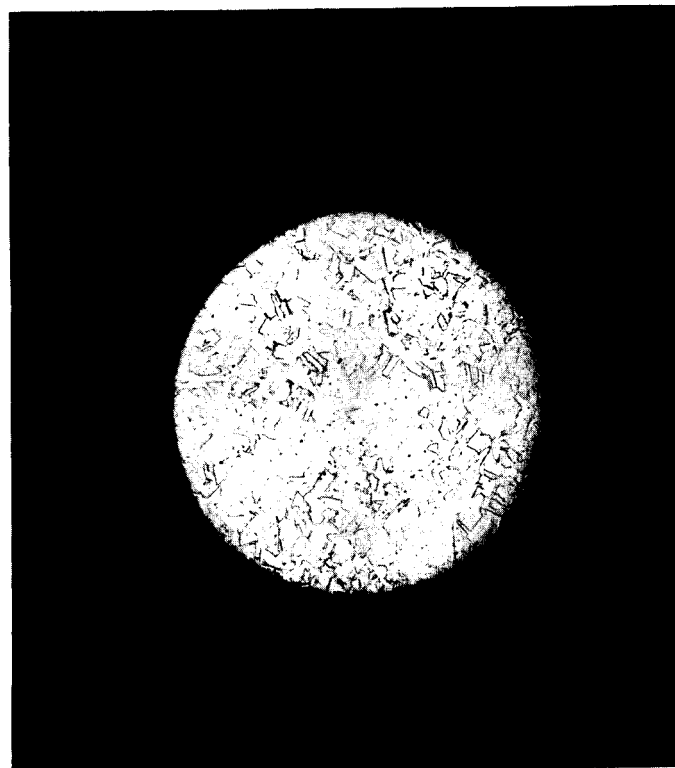
0.034" Dia.

7M796

100X

0.034" Dia.

7M798



100X

0.020" Dia.

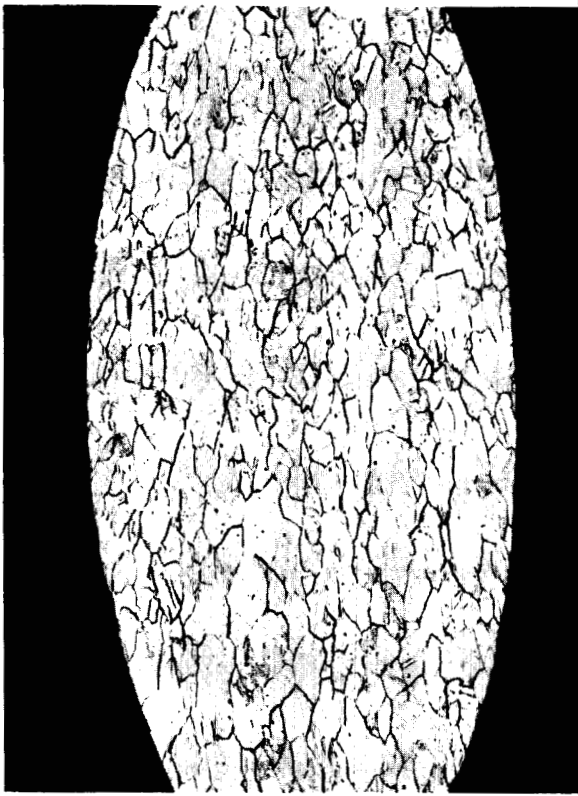
7M529

100X

0.020" Dia.

7M523

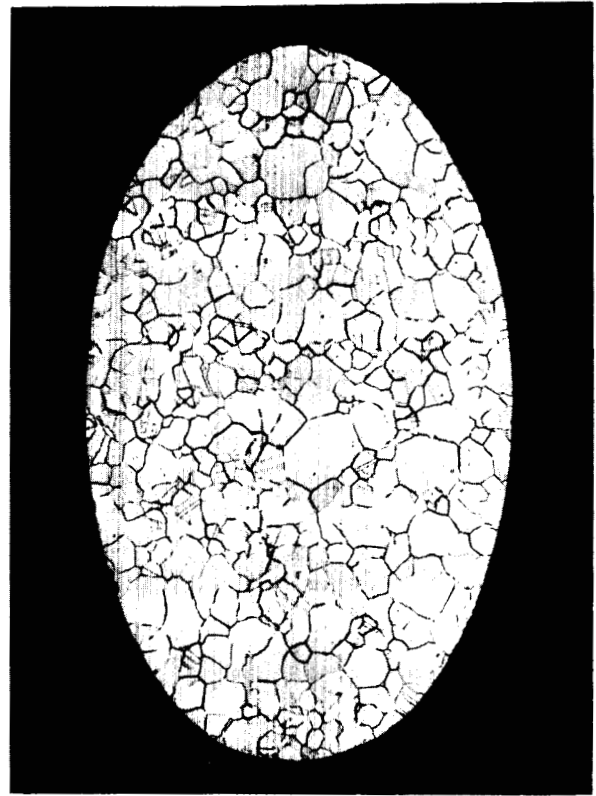
AS DRAWN



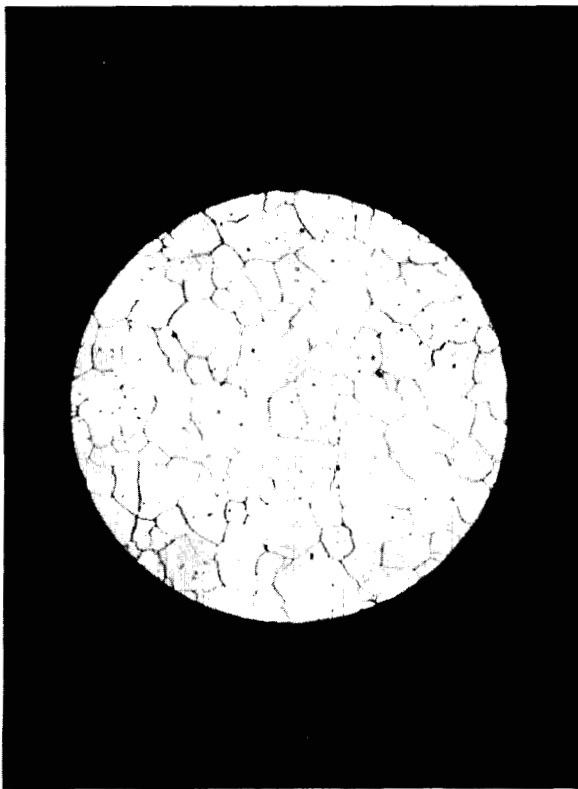
200X 0.011" Dia. 9M121

13

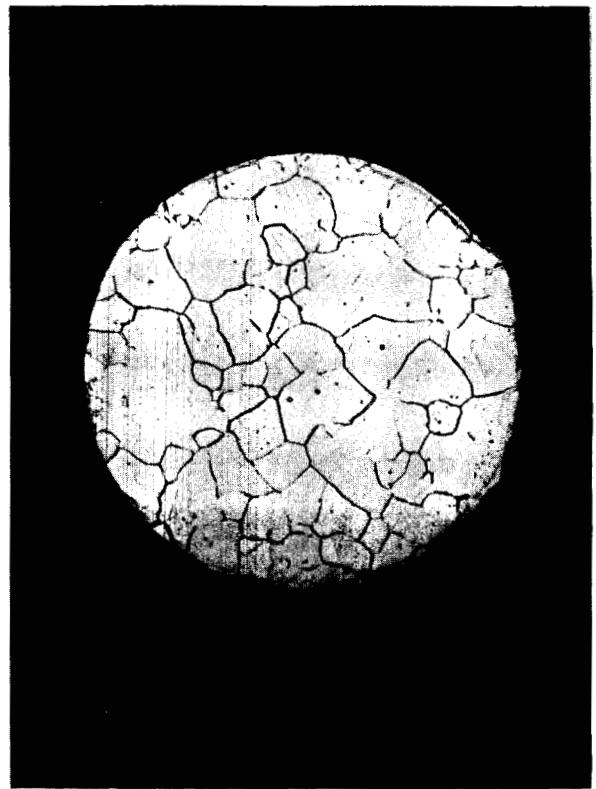
ANNEALED



200X 0.011" Dia. 9M118



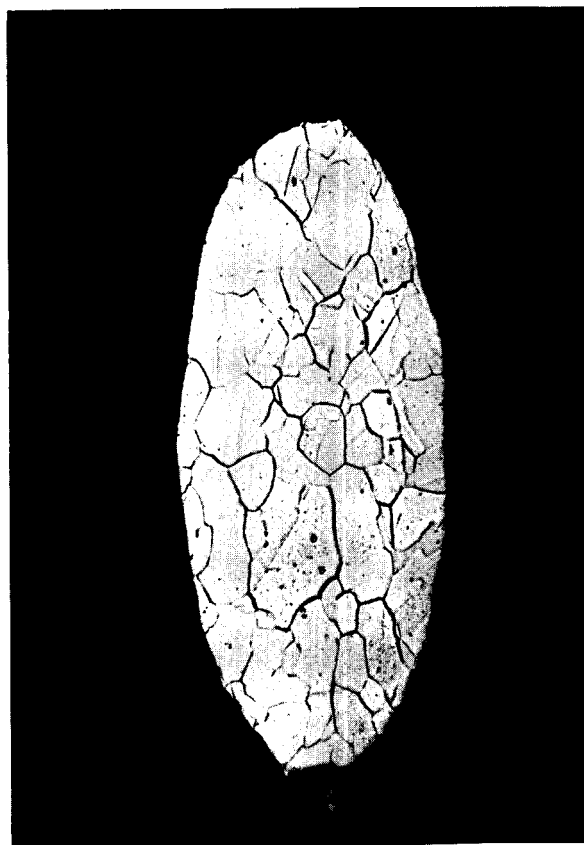
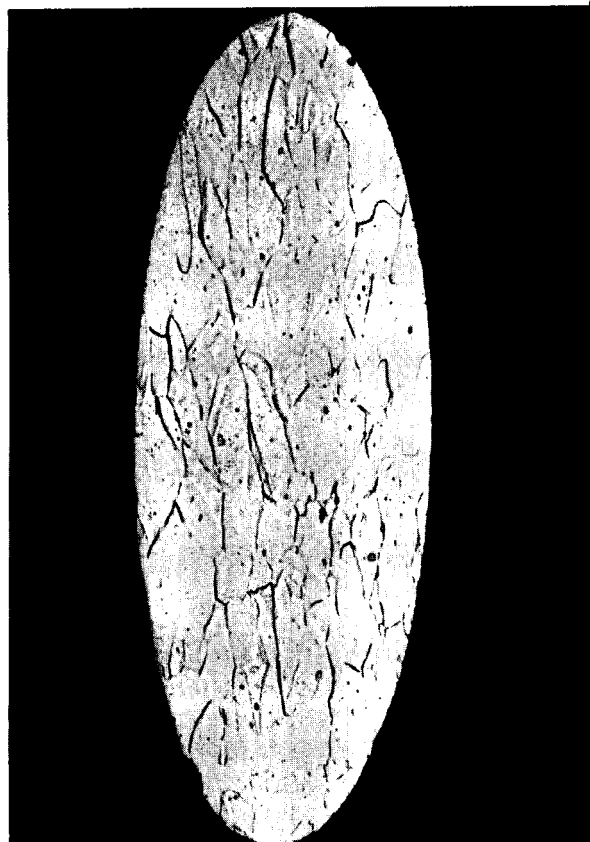
500X 0.0045" Dia. 9M149



500X 0.0045" Dia. 9M146

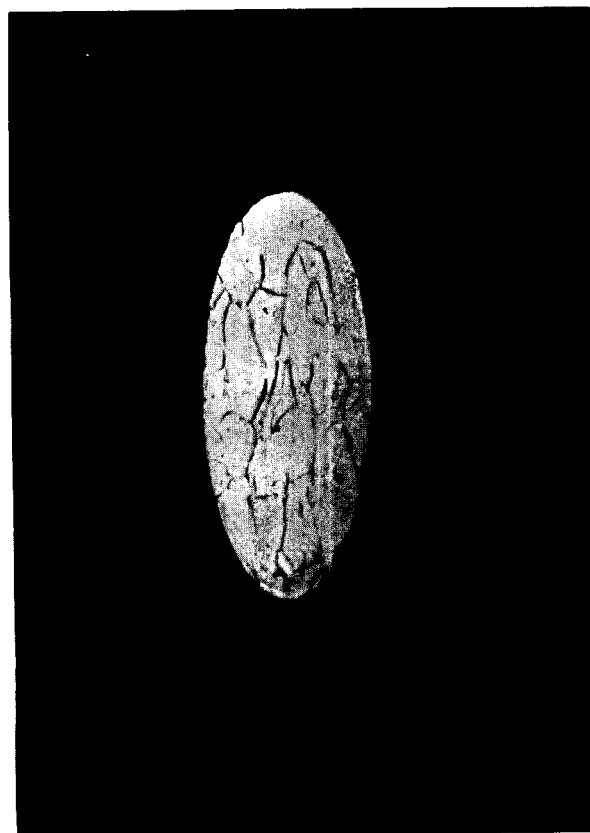
AS DRAWN

ANNEALED



500X 0.003" Dia. ON454

500X 0.003" Dia. ON455



500X 0.00176" Dia. ON456

500X 0.00176" Dia. ON457

APPENDIX B

M.M. LEMCOE

Consultant
(Recently Retired From Battelle)

Education

B.S., Civil Engineering, Washington University
M.S., Civil Engineering, Washington University
Ph.D., Civil Engineering, University of Illinois

Qualifications

Dr. Lemcoe specializes in high-temperature strain sensor technology, experimental stress analysis, mechanical and thermal fatigue analysis, and high-temperature structural behavior of materials. He has been actively involved in research relating to the conceptual design and development of very high temperature strain gages for use to at least 1900 F, and special strain sensors for use in instenst shock and ir-radiation environments. In so doing, he has extended the state-of-the-art of high-temperature strain measurement by nearly 1000 F. He recently completed several programs for NASA and the AEC related to the development of high temperature electric resistance strain gages.

He has over 20 years experience in a supervisory, program management, or project engineer capacity in multidisciplinary research and development activities within his fields of specialization. During this period, he has 2/4 of a million dollars. Principal sponsors have included NASA, AEC, and the Defense Nuclear Agency.

He is a recognized authority in the field of high temperature strain sensors and has written numerous papers on the subject. He is past chairman of the Society for Experimental Stress Analysis (SESA), Technical Committee on Strain Gages, past Delegate of Western Regional Strain Gage Committee, and is in the process of preparing a SESA Monograph on High Temperature Strain Gages. He is also Chairman of the Subcommittee on Plastic Fatigue Strength of the Pressure Vessel Research Committee of the Welding Research Council. This committee monitors research relating to fatigue, creep-fatigue interaction, cumulative damage, and fracture of welded joints and metals-on a world-wide basis, and is comprised of key technical people from government laboratories, industry and universities.

M. M. LEMCOE (Continued)Prior Professional Experience

Prior to coming to Battelle, he was associated with the North American Rockwell Corporation's Liquid Metal Engineering Center, and served on the Division Director's senior technical staff as a consultant and project engineer in areas relating to high-temperature strain gage research, high-temperature fatigue, thermal ratcheting, and structural behavior of fast breeder reactor systems and components. Previously he served with North American Rockwell's Atomics International Division, and was supervisor of the Experimental Mechanics Unit. He also served as Principal Investigator on a long-term program involving the development of strain-measuring techniques for use in hostile environments up to 1400 F.

Earlier he was Manager of the Strength Analysis Section, Department of Structural Research, at Southwest Research Institute. He also taught at Washington University and served in the U.S. Air Force during World War II, where he was assigned to the Dynamics Branch of Aircraft Laboratory, Wright-Patterson Air Force Base. He also worked at Curtiss-Wright Corporation, Airplane Division, where he conducted stress and vibration analyses of structural components of military aircraft and directed ground and laboratory vibration testing.

The following is a list of his professional affiliations, awards, and selected publications.

Professional Awards and Committees

Chairman, Subcommittee of Plastic Fatigue Strength of Pressure Vessel Research Committee, Welding Research Council, (PVRC). Member of PVRC Fabrication Division; member of PVRC Main Committee.

Member of SESA Technical Committee on Strain Gages, SESA Subcommittee on Monographs, SESA Paper Review Committee, SESA Applications Committee.

Past Chairman of SESA Technical Committee on Strain Gages.

Past Chairman, Special Advisory Committee for the Building Research Advisory Board of the National Academy of Sciences.

Past Delegate to Western Regional Strain Gage Committee.

Past Reviewer and Indexer for Applied Mechanics Reviews, American Society of Mechanical Engineers.

17605 Community St.
Northridge, CA 91325

October 8, 1986

Dr. Sidney Diamond
Battelle Columbus Division
505 King Avenue
Columbus, OH 43201-2693

Dear Sid,

I have examined the five (5) samples of Pd-13Cr wire marked A, C, D, E, G, and I have the following comments at this time:

- (1) In the nominal .020 in. dia. size, the ductility of the wire appears satisfactory, based on bend tests.
- (2) Resistivity calculations show the resistivity of the wire in the nominal .020 in. dia. to be about 83 micro-ohm-cm. This corresponds to a resistivity of about 502 ohms per circular mil-ft.

It should be noted that pure Pt, which should have a resistivity similar to palladium, has a resistivity of about 14 micro-ohm-cm. It is evident that the 13% Cr significantly increases the resistance of the palladium. This is good in terms of properties of resistance strain gages.

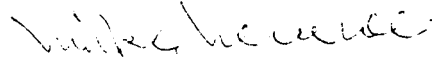
The resistivity of this alloy, however, appears to be somewhat lower than some of the other high-temperature strain gage alloys. Nichrome (80Ni-20Cr), for example, has a resistivity of about 675 ohms per circular mil-ft., and many of the Fe-Cr-Al alloys have a resistivity higher than 800 ohms per circular mil-ft.

In conclusion, it is my opinion, based on the ductility and resistivity properties at the .020 in. dia., that the alloy is sufficiently satisfactory, from the standpoint of ductility and resistivity, to justify drawing it down to strain gage wire size in the range of .001 to .002 in. in dia.

The submission of this letter report completes all work within the present scope of the effort.

Attached is the bill for these services.

Sincerely,



M. M. Lemcoe
Technical Consultant

MML:js

Enclosure (1)

BILLING INVOICE

1. Labor: 24 hours at \$45/hour	\$1,080.00
2. Expenses:	
Long Distance calls	\$ 9.60
Typing Services	<u>10.00</u>
	19.60
3. Total labor costs and expenses	<u>\$1,099.60</u>

Respectfully submitted,

M. M. Lemcoe

M. M. Lemcoe
Technical Consultant

17605 Community St.
Northridge, CA 91325

March 9, 1987

Dr. Sidney Diamond
Battelle Columbus Division
505 King Avenue
Columbus, OH 43201-2693

Dear Sid,

I have examined the nominal 1.7 mil Pd-Cr wire samples which you gave me with respect to resistivity properties and ductility--per our recent discussions. Calculations of resistivity shows only a small difference in resistivity between this diameter and the nominal .020 dia. (The 1.7 mil dia. wire has a resistivity of approximately 74 micro-ohm-cm. The .020 dia. wire has a resistivity of 83 micro-ohm-cm.) This difference is not considered significant and may well be within the measurement error limits.

The ductility appears very good, and I see no reason at this time why difficulties should be encountered in winding gages with this wire.

The submission of this letter report completes all work within the present scope of the effort.

Attached is the bill for these services.

Sincerely,



M. M. Lemcoe
Technical Consultant

MML:js

Enclosure (1)

APPENDIX E

INVENTORY OF VARIOUS FORMS OF 87%Pd-13%Cr ALLOY
DELIVERED TO NASA LEWIS RESEARCH CENTER

Contents of box

1.	package of scrap from casting							
2.	envelope of rod material (crack)							
3.	package of virgin material							
4.	package containing approx. 1720 ft. 0.003 annealed wire (damaged)							
5.	one spool 0.003 annealed wire approx. 193 inches long							
6.	" " " " " "					1915	"	"
7.	" " " " " "					123	"	"
8.	" " 0.00176 " " "					129	"	"
9.	" " " " " "					557	"	"
10.	" " " " " "					575	"	"
11.	" " 0.020 " " "					144	"	"
12.	" " 0.015 " " "					90	"	"
13.	" " 0.110 " " "					100	"	"
14.	" " 0.008 " " "					200	"	"
15.	" " 0.0059 " " "					450	"	"
16.	" " 0.0045 " " "					757	"	"

1. Report No. NASA CR-182304		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Fabrication of Pd-Cr Wire			5. Report Date July 1989		
			6. Performing Organization Code		
7. Author(s) Sidney Diamond and Dennen M. Leach			8. Performing Organization Report No. None		
			10. Work Unit No. 505-62-01		
9. Performing Organization Name and Address Battelle Columbus Division 505 King Avenue Columbus, Ohio 43201			11. Contract or Grant No. C-02999-J		
			13. Type of Report and Period Covered Contractor Report Final		
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191			14. Sponsoring Agency Code		
			15. Supplementary Notes Project Manager, Aloysius F. Hepp, Instrumentation Controls Technology Division, NASA Lewis Research Center.		
16. Abstract <p>Discussions among the staff of NASA Lewis Research Center, Battelle personnel, and Dr. M.M. Lemcoe indicated that NASA LeRC was interested in having a Pd-Cr alloy fabricated into fine wire. Battelle's experience in casting, metal working, and wire drawing were considered to be applicable to NASA LeRC's goal and Battelle submitted a letter proposal to melt, cast, and draw a specified alloy of palladium and chromium to wire of about 0.02-inch-diameter and to determine the drawing conditions for the material. It was understood by BCD that, if the condition of the material were to be judged by NASA LeRC to be acceptable at this point, a follow-on effort could be authorized at BCD to attempt to draw the PD-Cr alloy to very small diameter wire (0.003 and 0.001 inch). Both efforts were authorized by NASA and completed by BCD, with some modification in the final wire diameter. The wire produced by BCD in the two (related) programs has previously been sent to NASA Lewis. This report provides a description of the processing conditions and characterization of the material at various stages of the work.</p>					
17. Key Words (Suggested by Author(s)) Precious metal fabrication Vacuum annealing Palladium Palladium alloy			18. Distribution Statement Unclassified - Unlimited Subject Category 26		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No of pages 27	22. Price*