Naval Surface Warfare Center
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NSWCCD-61-TR—1998/25 **October 1998**

Survivability, Structures, and Materials Directorate
Technical Report

Spray Formed Ni-Cr Alloys for Shipboard Waste Incinerators

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NSWCCD-61-TR—1998/25 Spray Formed Ni-Cr Alloys for Shipboard Waste Incinerators

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED Research and Development

4. TITLE AND SUBTITLE Spray Formed Ni-Cr Alloys for Shipboard Waste Incinerators	5. FUNDING NUMBERS 98-1-6130-130 98-1-6120-780
6. AUTHOR(S) Leslie K. Kohler, Dr. Louis F. Aprigliano, Craig J. Madden	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Surface Warfare Center, Carderock Division Code 612, Materials Processing Branch West Bethesda, Maryland 20817	8. PERFORMING ORGANIZATION REPORT NUMBER NSWCCD 61-TR-1998/25
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Dr. Lawrence Kabacoff and Dr. John Sedriks Office of Naval Research 800 North Quincy Avenue Arlington, VA 22217	10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES
Program Element No. 0602234N
Project No. 03450 Task No. 98PR02611-00

12a. DISTRIBUTION/AVAILABILITY STATEMENT Distribution unlimited. Approved for public release.	12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The Naval Surface Warfare Center, Carderock Division, (NSWCCD) has produced several spray formed Ni-Cr alloys in a research effort to identify a replacement for conventionally processed Alloy 690 (60Ni-30Cr-9.5Fe) waste incinerator liners. Six alloys with chromium contents ranging from 37 to 50 weight percent were chosen for evaluation; 61.5Ni-37Cr-1.5Nb, 55.5Ni-43Cr-1.5Zr, 55.5Ni-43Cr-1.5Nb, Alloy 671 (52Ni-48Cr), 48.5Ni-50Cr-1.5Nb, and 50Ni-50Cr. These spray formed materials were characterized microstructurally and evaluated for strength, ductility, and hot workability. These measurements were compared with both conventional and spray formed Alloy 690 values.

Room and high temperature tensile properties showed that as-sprayed Ni-Cr alloys with chromium contents of 43% or greater exhibited mechanical properties that were equivalent to or better than typical values for wrought Alloy 690. This has identified these materials as candidates for corrosion testing and possible replacement of Alloy 690. It is expected that higher chromium contents will improve corrosion resistance.

Hot rolling of the spray formed high chromium alloys was performed easily under laboratory conditions, showing that these alloys in the spray formed condition can endure post processing without brittle cracking problems. This hot working improved the mechanical properties to values that exceeded their conventional wrought counterparts for both strength and ductility. This shows that any of the alloys in their spray formed and hot worked condition can be considered as possible replacements for Alloy 690, and should be subjected to corrosion tests.

14. SUBJECT TERMS Spray forming, Nickel, Chromium, Niobium, Zirconium, Alloy 690, Alloy 671, Nickel Alloys, Incinerator, Corrosion resistant, High temperature	15. NUMBER OF PAGES 17
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT Unclassified

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ABSTRACT

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Room and high temperature tensile properties showed that as-sprayed Ni-Cr alloys with chromium contents of 43% or greater exhibited mechanical properties that were equivalent to or better than typical values for wrought Alloy 690. This has identified these materials as candidates for corrosion testing and possible replacement for Alloy 690. It is expected that higher chromium contents will improve corrosion resistance.

Hot rolling of the spray formed high chromium alloys was performed easily under laboratory conditions, showing that these alloys in the spray formed condition can endure post processing without brittle cracking problems. This hot working improved the mechanical properties to values that exceeded their conventional wrought counterparts for both strength and ductility. This shows that any of the alloys in their spray formed and hot worked condition can be considered as possible replacements of Alloy 690, and should be subjected to corrosion tests.

ADMINISTRATIVE INFORMATION

This program has been funded by the Office of Naval Research, project number 03450. Dr. J. Sedricks and Dr. L. Kabacoff have been the ONR project monitors. The Principal Investigator for the program "High Chromium Alloys for Shipboard Waste Incinerators" is Robert L. Clarke of NSWCCD Code 613. This effort was performed by the Materials Processing Branch, NSWCCD Code 612 under Program Element 0602234N, and was supervised by the Branch Head, Dr. Louis Aprigliano.

ACKNOWLEDGMENTS

The significant technical assistance of Robert Mattox and Steven Szpara of the Spray Forming Group at NSWCCD is appreciated, as well as Albert Brandemarte of the Metallography Laboratory. Richard Rebis, now at Corning Inc., also contributed to the earlier Ni-Cr spray forming work.

INTRODUCTION

The US Navy uses shipboard incinerators for disposal of blackwater, and would like to build a multifunctional incinerator for disposal of a wider range of waste. The liners of the current incinerators utilize Alloy 690 (Ni-30Cr-9.5Fe)*, which limits the operating temperature to 700°C (1292°F) or less. Higher temperatures are required for a multi-functional shipboard incinerator. Increasing the chromium content of alloys is known to increase the operational temperature in many corrosive high temperature environments.[1,2] However, alloys containing more than 30% Cr become increasingly difficult to produce by hot working because of a brittle alpha chromium phase.[3] The current manufacturing process for liners requires a cast alloy to be rolled into sheet form, then bent and welded into a final tubular shape. Alloy 671 (52Ni-48Cr) is an example of a high Cr alloy that can be formed, but requires several intermediate anneals in order to avoid work hardening and cracking.[4] A 1986 technical report shows that Alloy 671 was actually the material of choice instead of Alloy 690 for incinerator liners, but production of Alloy 671 plate had been discontinued by the International Nickel Co., Inc. (INCO).[5] In light of these facts, a program was initiated with the goal of producing fabricable, high chromium, nickel based alloys capable of operating at 980°C (1796°F) in the corrosive wastewater environment.

The production method chosen for this research is metal spray forming. Successes in spray forming of nickel based alloys, such as Inconels 625 and 718, are well documented.[6,7] Metal spray

* All compositions are listed in weight percent

forming, also called the Osprey process, is a three step process combined into one operation. These steps are melting, atomization and deposition. More specifically, when a stream of the molten alloy is allowed to exit a crucible, it is atomized into a spray using high pressure gas. This semisolid spray is collected by a substrate, where the deposit will continue to solidify and build to a desired near net shape. Classic advantages of spray forming include reductions in processing time and costs due to the one step nature of the process and its near net shape result. There are also benefits in the microstructure, which consists of fine equiaxed grains and very little segregation. Many spray formed alloys have strengths which are equal to or better than their conventionally cast and wrought counterparts. Often, these alloys still need minimal secondary processing and/or machining, but the grain refined microstructure lends itself to better workability and machinability.[7,8]

Six Ni-Cr alloys with chromium contents ranging from 37 to 50 weight percent were chosen and spray formed for this study, as well as Alloy 690 (30%Cr). Three of the chosen alloys contained niobium, which has been shown to improve high temperature strength and thermal stability.[2,3] One alloy contained zirconium, which has been shown to increase ductility.[2,3]

If this program and its future corrosion tests prove to be successful, manufacturing is envisioned to take place at the Navy MANTECH spray forming plant located in Barberton, Ohio. This plant, the largest of its kind, has been designed and commissioned to produce large diameter tubulars. Incinerator liners fall easily into this category, with dimensions of 22.75 inch diameters and 0.25 wall thickness.

EXPERIMENTAL PROCEDURES

Spray forming was performed at the NSWCCD non-reactive pilot research facility using an 80 pound bottom pour melt system. Argon was used as the melt cover gas, while nitrogen was used as the scanning atomization gas. Mild steel tubes of 4 inches outer diameter and 0.065" wall thickness were used as substrates. A single pass motion plan was used to translate the rotating substrates beneath the spray. Final dimensions of the spray formed tubes were approximately six inches outer diameter (one inch wall thickness), and seven inches in length after end cropping.

Gas to metal ratio (GMR) is the primary parameter for predicting the temperature and stability of the spray and the deposit. In general, low GMR's correspond to hot deposits, low gas atomization pressures, and high metal flow rates. If a deposit is too hot, it will have poor structural integrity, low yields, and a rough surface. If a deposit is too cold, it will have low yields and increased porosity. Optimum GMR for these alloys proved to be between 0.45 and 0.6 for these runs. All runs were performed under these conditions, with the exception of the Zr containing alloy. No deliberate change was made for this alloy, yet it consistently exhibited a decreased metal flow rate. This in turn decreased the GMR and created a deposit that was too cold. Decreased metal flow rates in spray forming are normally attributed to a reaction of the alloy with refractory components of the system, or with the gas at the nozzle exit. These reactions can cause either an effective decrease in the nozzle bore, or an increase in melt viscosity. The Zr in this alloy is likely to be a source for these types of reactions.

Spray formed tubes were sectioned longitudinally in 1 inch wide strips for subsequent machining of specimens for tensile testing, hot rolling, and microstructural examination. All reported tensile properties are an average of three or more standard 0.252 inch gauge specimens. Samples for microstructural characterization were electrolytically etched using 10% oxalic acid. Area reductions of 2:1 were accomplished by hot rolling round bars of 0.8 inches diameter at 1200°C. Hot rolling successfully reduced all of the alloys.

RESULTS

MICROSTRUCTURES

In the solid state, Ni-Cr alloys with 30-50% Cr can have either a single γ Ni phase or a dual phase microstructure of γ Ni plus α Cr. A Ni-Cr phase diagram is shown in Figure 1[9], along with an expanded view of the eutectic region. The eutectic composition is 49Ni-51Cr, and a solubility limit of 47% chromium in nickel occurs at 1345°C (2453°F). Each of the alloys is marked on the simplified diagram to show if they are expected to solidify into the single or dual phase region. Three alloys of interest contain greater than 47% chromium and solidify in the dual phase region, while the other four alloys have less than 47% chromium and exhibit single phase structures.

Microstructures of each of the spray formed dual phase alloys are shown in Figures 2a through 2c. The dark regions represent the eutectic structure of α Cr plus γ Ni, while the surrounding area is γ Ni. It can be seen that as the chromium content increases the amount of second phase α Cr increases. Figure 2d is a higher magnification view of the second phase regions, which have a lamellar type of structure.

Microstructures of the four single phase alloys are shown in Figures 3a through 3d. Grain sizes of the first three alloys are very similar, averaging between 40 and 50 microns. The fourth alloy is the Zr containing alloy with an average grain size of 15 to 20 microns. These smaller grains, as well as some layering effects, are a result of the colder spray forming conditions that were described in the Experimental Procedures section.

Representative microstructures of two hot rolled alloys, Alloy 690 and Alloy 671, are shown in Figures 4a and b. Both alloys experienced a reduction in grain size to near 20μm. The Cr rich regions in the dual phase alloy are also smaller and more dispersed. At this magnification, the lamellar features of the eutectic structure are no longer visible as they were in the as-sprayed dual phase alloys.

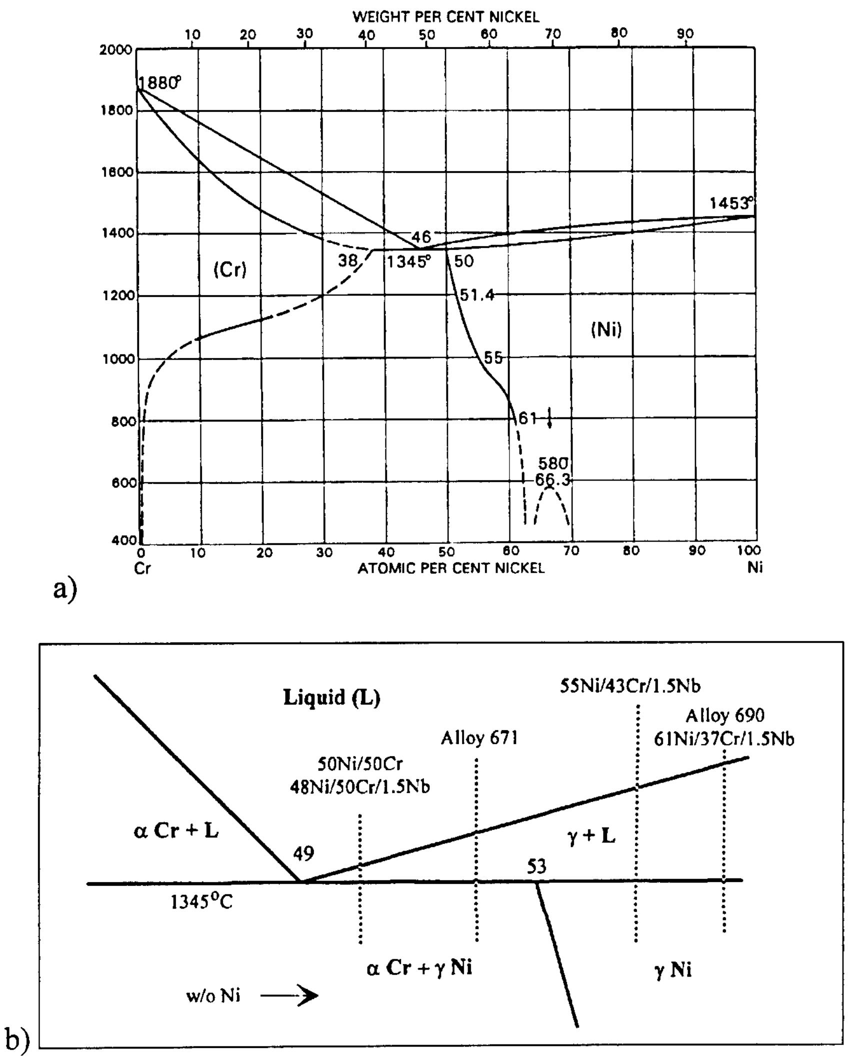
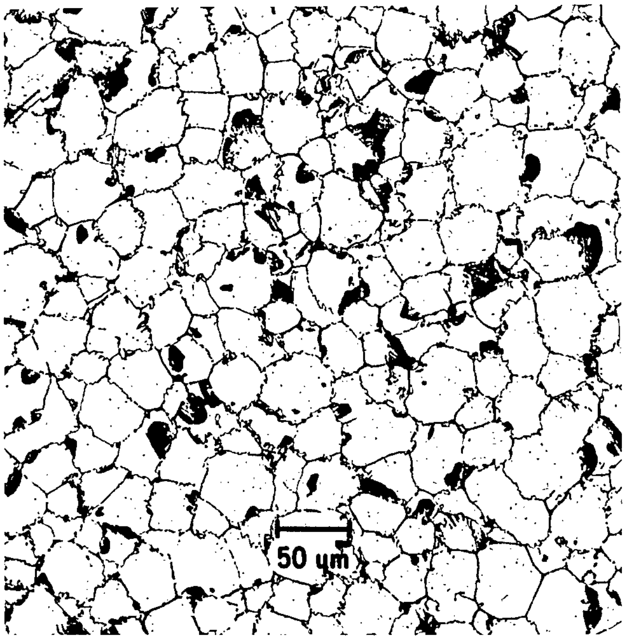
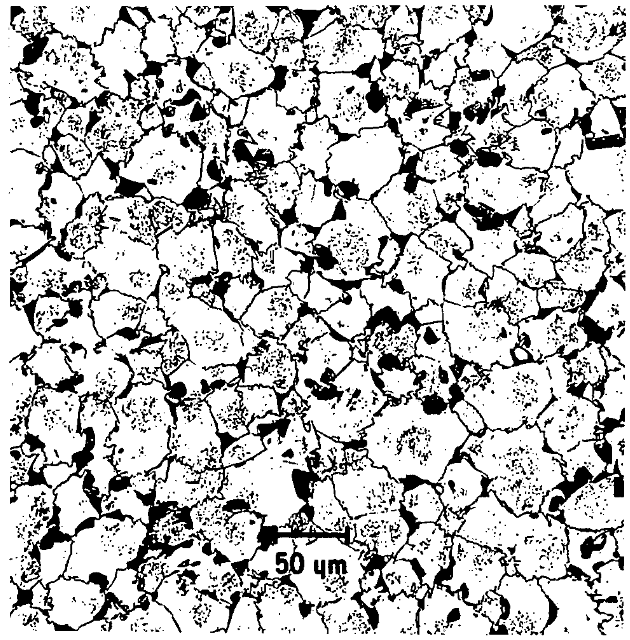


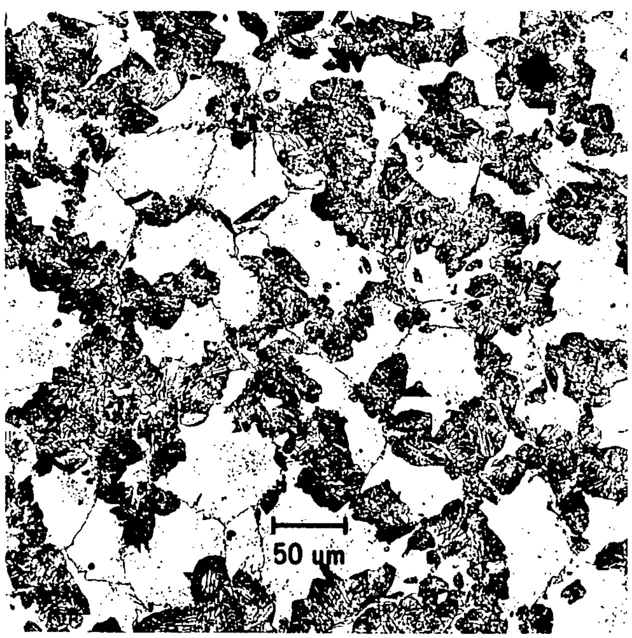
Figure 1: Ni-Cr Phase Diagram(9) a) Full scale b) Region of interest.



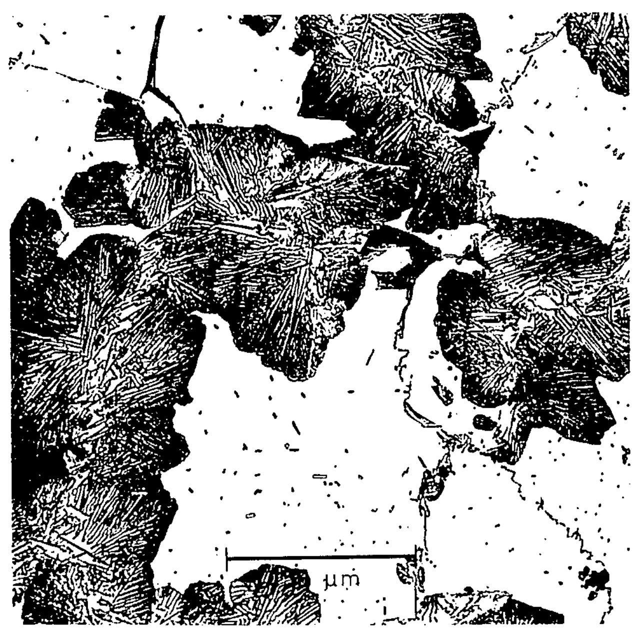
a) Alloy 671 (52Ni-48Cr), 200x



b) 48.5Ni-50Cr-1.5Nb, 200x

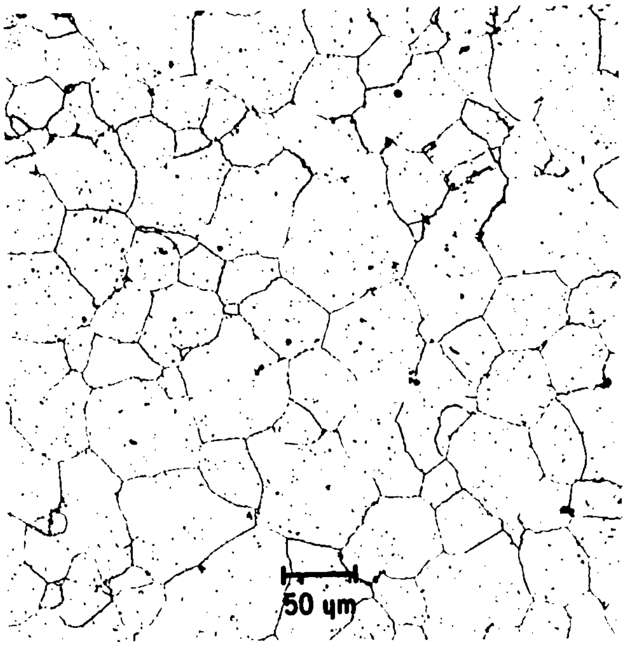


c) 50Ni-50Cr, 200x

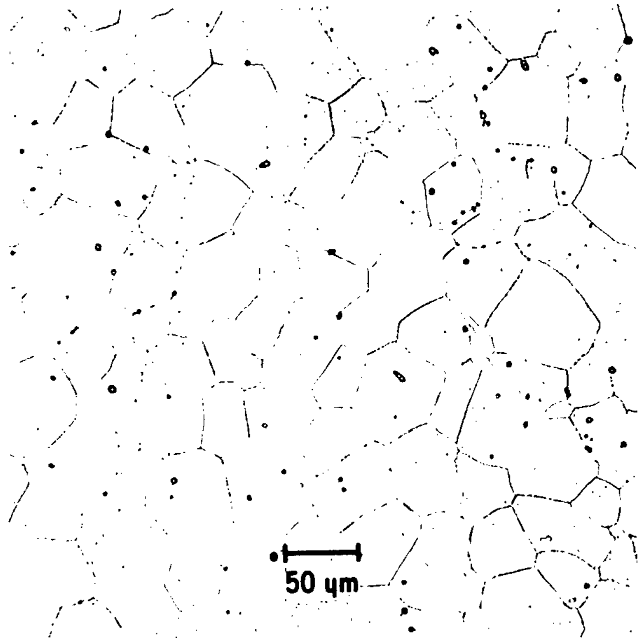


d) 50Ni-50Cr, Cr rich regions, 500x

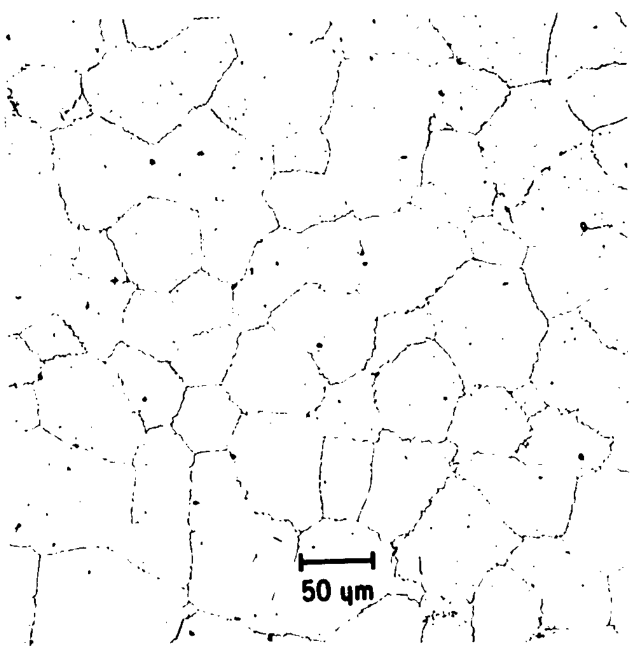
Figure 2: Optical micrographs of dual phase Ni-Cr alloys. Etched – 10% oxalic acid.



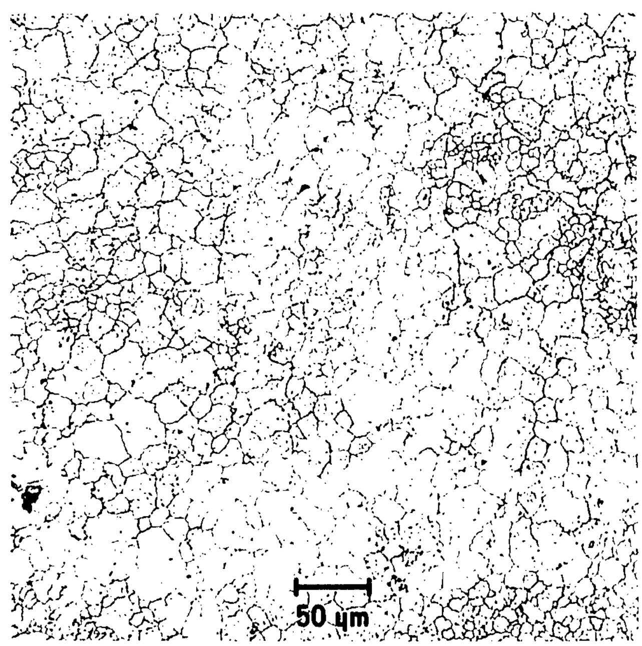
a) Alloy 690 (60Ni-30Cr-9.5Fe), 200x



b) 61.5Ni-37Cr-1.5Nb, 200x

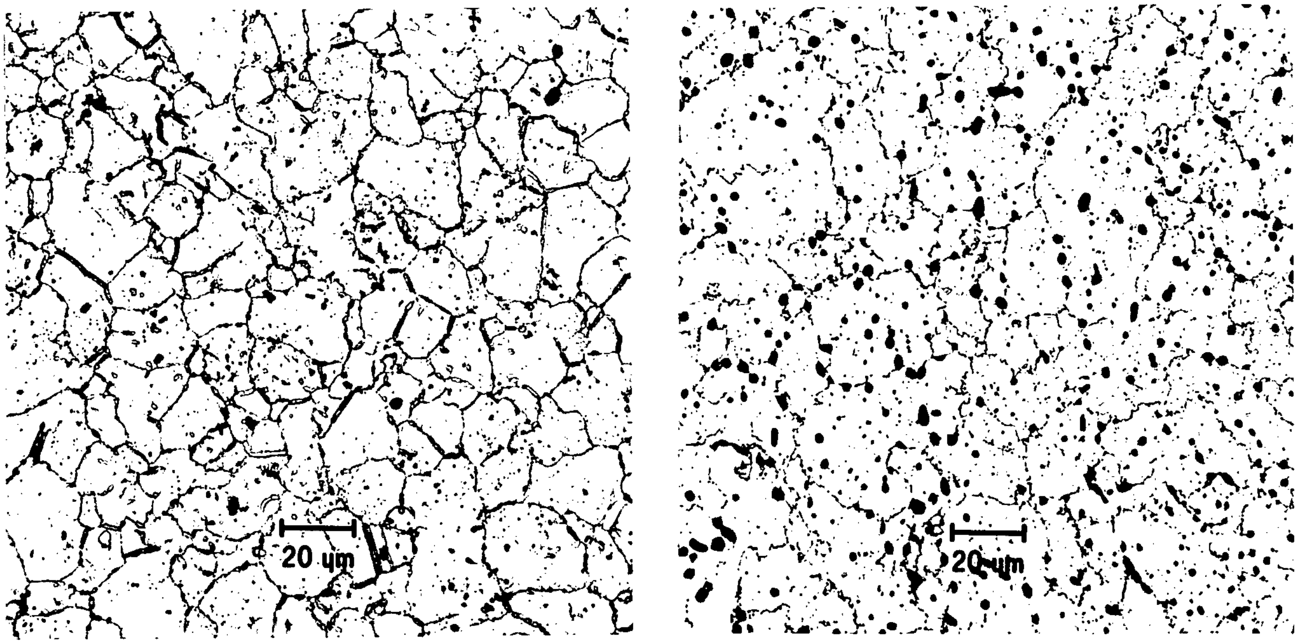


c) 55.5Ni-43Cr-1.5Nb, 200x



d)55.5Ni-43Cr-1.5Zr, 200x

Figure 3: Optical micrographs of single phase Ni-Cr alloys. Etched – 10% oxalic acid.



a)Alloy 690, 500x b) Alloy 671, 500x

Figure 4: Optical micrographs of spray formed and hot rolled Ni-Cr alloys. Etched – 10% oxalic acid.

MECHANICAL PROPERTIES

Tensile properties of the alloys in the as-sprayed condition were measured at room temperature and are listed in Table 1. In general, the higher chromium containing alloys exhibited higher tensile strengths and lower ductility. This is due to the greater presence of the brittle alpha chromium phase. 50Ni - 50Cr had the highest room temperature yield and ultimate tensile strengths of 987 MPa and 1283 MPa respectively, while Alloy 690 had the lowest values of 276 MPa and 635 MPa.

The highest elongation values were 56% and 57% for Alloys 690 and 55.5Ni-43Cr-1.5Nb respectively, while 55.5Ni-43Cr-1.5Zr exhibits the lowest elongation, 21%. The Zr addition was expected to increase the ductility[2,3], but porosity from the colder conditions of the run counteracted any effects that the Zr may have had.

Table I: Room Temperature Mechanical Properties of Spray Formed Ni-Cr Alloys

As Sprayed Alloy	Room Temperature		
	UTS MPa (ksi)	0.2% YS MPa (ksi)	% Elongation
690 (60Ni-30Cr-9.5Fe)	635 (92)	276 (40)	56
61.5Ni-37Cr-1.5Nb	676 (98)	331 (48)	46
55.5Ni-43Cr-1.5Zr	697 (101)	366 (53)	21
55.5Ni-43Cr-1.5Nb	752 (109)	400 (58)	57
671 (52Ni-48Cr)	773 (112)	386 (56)	38
48.5Ni-50Cr-1.5Nb	1035 (150)	614 (89)	32
50Ni - 50Cr	1283 (186)	987 (143)	33
Conventional Alloy			
Alloy 690 ASME Code(10)	552 (80)	276 to 448 (40 to 65)	30
Alloy 690(11)	731 (106)	365 (53)	41
Alloy 671(11)	862 (125)	483 (70)	25
Uniloy 49Ni-50Cr-1Ti(12)	918 (133)	552 (80)	18

As expected, the next lowest ductility values come from the highest Cr containing alloys. However, these values are still very high for dual phase alloys. This fact is displayed in Figure 5, a plot of elongation % as a function of chromium content. The curved line represents conventionally cast alloys while the individual markers represent the values of spray formed alloys. Spray formed single phase alloys tend to have less ductility than the cast equivalent, while spray formed dual phase alloys have greater ductility.

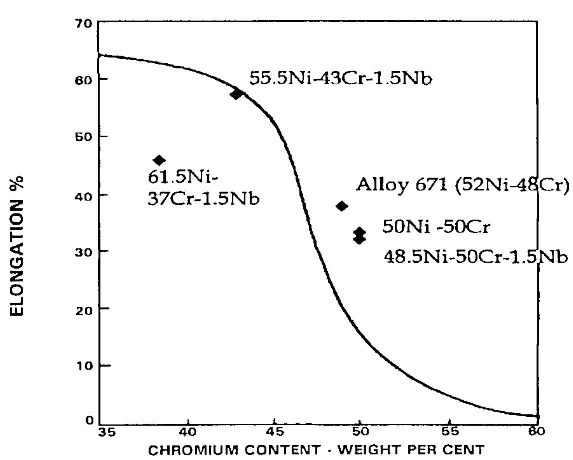


Figure 5: Elongation vs. Cr content for cast Ni-Cr.[13] Extra data points represent spray formed values.

Table I also includes several standard wrought mechanical property values for comparison. These include the Alloy 690 ASME Boiler and Pressure Vessel Code, and typical values for conventionally processed Alloys 690, 671, and Uniloy 49Ni-50Cr-1Ti. All of the as-sprayed alloys meet the minimum specifications of the ASME code. There is also a maximum yield strength specification of 448MPa in this code that the two spray formed alloys with 50% Cr exceed. Despite this, these two alloys are exceptional, as they have higher values than the Uniloy alloy for both strength *and* ductility. Strengths of spray formed Alloy 690 and spray formed Alloy 671 are below that of their wrought counterparts, while their elongations are higher.

Table II: Tensile Properties of Spray Formed plus Hot Rolled Ni-Cr Alloys (2:1 area reduction)

Spray Formed Alloy After 2:1 Area Reduction	Room Temperature		
	UTS MPa (ksi)	0.2% YS MPa (ksi)	% Elongation
Alloy 690 (60Ni-30Cr-9.5Fe)	766 (111)	538 (78)	48
61.5Ni-37Cr-1.5Nb	842 (122)	545 (79)	42
55.5Ni-43Cr-1.5Zr	883 (128)	628 (91)	32
55.5Ni-43Cr-1.5Nb	883 (128)	593 (86)	48
671 (52Ni-48Cr)	987 (143)	718 (104)	35
48.5Ni-50Cr-1.5Nb	1242 (180)	918 (133)	21
50Ni – 50Cr	1194 (173)	897 (130)	33
Conventional Alloy			
Alloy 690 ASME Code(10)	552 (80)	276 to 448 (40 to 65)	30
Alloy 690(11)	731 (106)	365 (53)	41
Alloy 671(11)	862 (125)	483 (70)	25
Uniloy 49Ni-50Cr-1Ti(12)	918 (133)	552 (80)	18

The strengths of these Alloys 690 and 671 were greatly improved by hot rolling. The tensile properties for spray formed materials hot rolled with a 2:1 area reduction are shown in Table II. In general, the hot worked alloys display higher strengths and lower ductility than they did in their as-sprayed condition. One exception is the Zr containing alloy, in which the porosity was decreased by hot rolling, and therefore the elongation value rose to a more acceptable level. Another exception is the decrease in strengths for the 50Ni-50Cr alloy, but the reason for this change is unknown. Alloys 690 and 671 had enough increase in strength values to now exceed typical wrought values. Furthermore, their percent elongations have decreased slightly, but still remain above those of wrought standards. All of the hot rolled alloys had yield strengths that exceeded the maximum specification for the ASME Boiler and Pressure Vessel Code. If a certain application required these types of properties, it is expected that annealing of these hot worked alloys would reduce the yield strength.

High temperature mechanical properties measured for three of the spray formed alloys are listed in Table III. Niobium was added to these alloys based on studies which have shown that nitride forming elements improve workability as well as high temperature strength and ductility.[14] It is difficult to isolate the effect of Nb since there were no spray formed alloys without additions tested in this high temperature region. Without making any conclusions about these effects, it is noted that the 55.5Ni-43Cr-1.5Nb shows very high elongations at both temperatures, and 48.5Ni-50Cr-1.5Nb has strengths which are far superior to those of both the Uniloy alloy and Alloy 671 (52Ni-48Cr).

Table III: High Temperature Mechanical Properties of Ni-Cr Alloys

As Sprayed Alloys	482°C (900°F)			649°C (1200°F)		
	UTS MPa (ksi)	0.2% YS MPa (ksi)	% Elongation	UTS MPa (ksi)	0.2% YS MPa (ksi)	% Elongation
61.5Ni-37Cr-1.5Nb	511 (74)	186 (27)	52	435 (63)	186 (27)	42
55.5Ni-43Cr-1.5Zr	504 (73)	269 (39)	18	428 (62)	235 (34)	15
55.5Ni-43Cr-1.5Nb	573 (83)	242 (35)	66	504 (73)	228 (33)	60
48.5Ni-50Cr-1.5Nb	828 (120)	497 (72)	39	704 (102)	455 (66)	31
Conventional Alloys						
Alloy 690[11]	587 (85)	262 (38)	45			
Alloy 671[11]	690 (100)	311 (45)		587 (85)	276 (40)	
Uniloy 49Ni-50Cr-1Ti[12]	690 (100)	400 (58)		552 (80)	400 (58)	

DISCUSSION

A wide range of high chromium nickel based alloys have been spray formed, and in some cases post processed, and evaluated with respect to their mechanical properties. The most dramatic improvement in tensile properties was exhibited by two spray formed alloys with 50% Cr. These had significantly greater strengths and ductility than the closest standard alloy comparison of Uniloy 49Ni-50Cr-1Ti. Alloy 690 and 671 showed similar improvements over standard material when spray formed and hot rolled. These are excellent results, and it is possible that these alloys could be exploited in Naval applications beyond incinerators.

Since there are currently no strength specifications for incinerator liners, the best reference point for choosing alloys for further corrosion testing will be the strength values of current Alloy 690 liners. All seven of the spray formed plus hot rolled alloys have strengths which are better than standard Alloy 690, and therefore should be further tested as possible replacements of Alloy 690. Even more cost effective, though, are the spray formed alloys which do not need further processing to increase strength. As-sprayed alloys with 43% Cr or greater also have strengths which are equivalent to or better than conventional Alloy 690. The higher Cr containing alloys are also expected to have better corrosion performance, as will be verified in future tests.

Although this study was done with multi-functional incinerators in mind, it is possible that spray forming can provide significant cost savings if it is also used for manufacturing the current blackwater incinerator liners. As mentioned in the introduction and in Navy reports, if Alloy 671 had been commercially available in this product form, it would be used instead of Alloy 690. Burner-rig tests showed that Alloy 671 had better hot corrosion resistance than several different alloys, including Alloy

690.[15,16] A full scale prototype of Alloy 671 showed a life of 1700 hours, with failure due to cracking instead of hot corrosion.* It is possible that the inherent ductility of spray formed Ni-Cr alloys would significantly extend the service time for Alloy 671 and other high Cr alloys. Less frequent replacement of a higher Cr alloy liner combined with near net shape processing creates the basis for potential life cycle cost savings in any type of incinerator liner. This leads to the recommendation that spray formed Ni-Cr alloys with 43% chromium content or greater be considered as replacements for Alloy 690 incinerator liners.

CONCLUSIONS

- Ni-Cr alloys having Cr contents between 30% and 50% were successfully spray formed.
- Hot rolling to a 2:1 area reduction was successful for spray formed high chromium alloys.
- Zr, added to a Ni-Cr alloy to increase ductility, caused unpredictable behavior in spray forming, resulting in reduced metal flow rates and porosity in the finished product.
- Spray formed alloys with Cr contents of 43% or below had single phase microstructures, while Cr contents of 48% or above resulted in duplex microstructures of gamma nickel and gamma nickel-alpha chromium eutectic.
- Spray formed plus hot rolled Ni-Cr alloys had both strength *and* elongation values that were greater than standard wrought alloys of similar composition.
- Tensile properties of as-sprayed Ni-Cr alloys with chromium contents of 43% to 50% were equal to or better than average values of conventional Alloy 690.
- As-sprayed Ni-Cr alloys with 43% chromium or greater are the best candidates for replacement of Alloy 690 incinerator liners, both blackwater and multi-functional. This conclusion is based upon

* Yerkovich, L.A., Huntington Alloys Ltr K-1 to NAVSESS DET (8 Feb 1980)

their tensile properties and the potential cost savings of spray forming as a near net shape process. It is anticipated that the alloys with the highest chromium contents will also have the best performance in future corrosion tests.

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