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# STUDY OF HIGH PERFORMANCE ALLOY ELECTROFORMING

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# STUDY OF HIGH PERFORMANCE ALLOY ELECTROFORMING

## ABSTRACT

Nickel-manganese alloy electrodeposits from an electrolyte containing more manganese ion than previously used is being evaluated at two bath operating temperatures with a great variety of pulse plating conditions. Saccharine was added as a stress reducing agent for the electroforming of several of the samples with highest manganese content. All specimens for mechanical property testing have been produced but are not through the various heat treatments as yet. One of the heat treatments will be at 343°C (650°F), the temperature at which the MCC outer electroformed nickel shell is stress relieved. A number of retainer specimens from prior work have been tested for hardness before and after heat treatment. There appears to be a fairly good correlation between hardness and mechanical properties. Comparison of representative mechanical properties with hardnesses are made for nickel-manganese electrodeposits and nickel-cobalt-manganese deposits.

## I. INTRODUCTION

The purpose of this work is to develop and demonstrate a system for electroforming materials with improved strength and high-temperature properties. The Space Shuttle Main Engine employs a main combustion chamber (MCC) where final combustion of propellant at high temperature and pressure takes place. This critical component must be structurally supported by a nickel-base alloy jacket. Producing this jacket from formed wrought metal segments requires numerous weldments which alter the mechanical properties of the base metal through heat affected zones. This requires thickening the alloy where joints are to be made to meet the structural requirements of the shroud. The use of electroformable alloys with great strength would have the potential for simplifying fabrication procedures for structural jackets and reducing overall weight by removing weldments. Such an electroformable alloy might also afford a possible use in advanced engines where light weight and good strength at high temperatures are necessary.

## II. TECHNICAL PROGRESS SUMMARY

### A. Task I - Literature Survey (Phase A)

The draft has been reviewed and approved. Any decision to publish additional copies will be made at the end of the contractual work effort by mutual agreement. This task is now considered complete.

### B. Task II - Alloy Characterization and Optimization (Phase A)

Ten new double panels of nickel-manganese electroformed alloy were produced from the 160 liter bath employing 30 percent more manganese ion in the electrolyte than previously used. Electroforming parameters (pulsing) were varied in such a series of procedures so as to provide alloy manganese contents ranging from 0.10 to 0.68 percent by weight. Attempts to increase the manganese content further led to cracking problems in the alloy due to high stress. This could be overcome by adding minute amounts of sodium saccharin to the bath to reduce the tensile stress. Previous experience has shown that the slightly higher sulfur content in the alloy from saccharin does not lead to hot-shortness due to the fact that manganese chemically combines with the sulfur - but only after a heat treatment.

If one examines the mechanical property test data reported in the previous technical progress summary, it will be noted that Sample Numbers NMS-6 and NMS-7 exhibited better yield strength performance than Sample Number NM-15 up to a test temperature of 260°C (500°F) even though the latter alloy contained more manganese. The NMS code for the former two samples indicates that saccharin was present in the bath as a stress reducer. Stress reducers normally contribute to finer grain size as well. This may be an important factor in producing extremely high mechanical and yield strengths. In reviewing all mechanical property test data on nickel-manganese alloys produced to data, the highest mechanical strengths for given manganese contents appear to be associated with those specimens produced from a bath containing modest amounts of saccharin. Test data from the series of nickel-manganese alloys now in heat treatment should confirm these findings and provide data by which we will determine if use of saccharine and reasonably moderate heat treatments will provide our final alloy selection which will be (a) low stressed, (b) strong as compared to Inconel 718, and (c) ductile enough for structural applications.

A large number of specimens of nickel-manganese and nickel-cobalt-manganese electrodeposits have been tested for mechanical properties before and after heat treatment. These have been tested for hardness against the Rockwell C scale - this scale having been selected on the basis that age-hardened Inconel 718 has a hardness of approximately Rockwell C 45. Table I provides mechanical property-hardness comparison data for nickel-manganese electroformed alloys. The samples are listed in order of increasing manganese content. While a general tendency for hardness to increase with manganese content is shown, there are obvious exceptions such as Sample Number NM-22 where a pulse plating duty cycle of only 12.5% was used. This tends to promote a much finer grain size which appears to be maintained through moderate heat treatments. Since all of the listed alloys contain rather fine grain sizes, the elongations shown for 2 inch gauge lengths may be misleading with regard to true ductility. Most elongation occurs in a fairly short gauge distance. We expect to see very satisfactory elongation results on round test bars to be made shortly. It is also significant to note that several specimens could not be mechanically tested due to cracking during machining. This is believed due to the fact that the machining of test strips was necessary prior to heat treating since various heat treatments were required with samples from a single electroformed panel.

It is interesting to note that none of the Rockwell C hardness values were as high as 45 as expected in age-hardened Inconel 718; however, many of the nickel-manganese alloys exhibited ultimate and yield strengths equivalent to the hardened Inconel control material.

Table II provides a similar comparison of mechanical property-hardness data for our experimental nickel-cobalt-manganese electroformed alloys. The elongation measurements are based on a 2 inch gauge length and do not reflect true ductility for reasons previously discussed with nickel-manganese alloys. The Rockwell C hardness values shown in both Table I and Table II are averages of three readings. The mechanical property data for nickel-cobalt-manganese

TABLE I - MECHANICAL PROPERTY/HARDNESS DATA FOR ELECTROFORMED NICKEL-MANGANESE ALLOYS

| Sample Number | Alloy Composition: Manganese (% by Wt.) | Alloy Composition: Sulfur (PPM) | Property Tested | Mechanical Property/Hardness Test Results at Ambient Temperature: As Deposited | Heat Treated 400°F(72 Hr) | Heat Treated 600°F(24 Hr) | Heat Treated 600°F(48 Hr) | Heat Treated 800°F(4 Hr) | Heat Treated 1000°F(2 Hr) |
|---|---|---|---|---|---|---|---|---|---|
| NM-21 | 0.018 | 10 | Ultimate (ksi) | 135.0 | 135.2 | 116.5 | | 80.7 | |
| | | | Yield (ksi) | 88.6 | 102.6 | 90.2 | | 60.5 | |
| | | | Elong.(%) | 10 | 7.8 | 12 | | 27.8 | |
| | | | Hardness ($R_c$) | 24.8 | 23.7 | 22.3 | | Below C Scale | |
| NM-22 | 0.103 | 8 | Ultimate (ksi) | 183.4 | 182.7 | 168.3 | | 142.3 | |
| | | | Yield (ksi) | 143.8 | 130.7 | 122.6 | | 114.1 | |
| | | | Elong.(%) | 5.6 | 6.5 | 15.0* | | 9.2 | |
| | | | Hardness ($R_c$) | 35.3 | 37.0 | 34.8 | | 29.7 | |
| NM-20 | 0.167 | 20 | Ultimate (ksi) | 148.4 | 156.0 | Sample cracked | | 154.9 | |
| | | | Yield (ksi) | 99.9 | 118.7 | in machining. | | 128.0 | |
| | | | Elong.(%) | 8.1 | Broke out of ga. | | | 5.2 | |
| | | | Hardness ($R_c$) | 27.8 | 31.3 | 33.0 | | 31.3 | |
| NM-13 | 0.188 | 19 | Ultimate (ksi) | 164.3 | 156.3 | 156.7 | | | 123.3 |
| | | | Yield (ksi) | 123.6 | 126.8 | 137.9 | | | 109.7 |
| | | | Elong.(%) | 7.0 | 6.0 | 5.0 | | | 7.0 |
| | | | Hardness ($R_c$) | 31.0 | 33.0 | 31.8 | | | 27.2 |
| NM-14 | 0.210 | 42 | Ultimate (ksi) | 149.5 | 148.4 | 154.7 | | 133.4 | |
| | | | Yield (ksi) | 102.2 | 115.3 | 124.5 | | 114.3 | |
| | | | Elong.(%) | 8.5 | 9.5 | 8.0 | | 9.0 | |
| | | | Hardness ($R_c$) | 30.0 | 30.5 | 32.3 | | 27.5 | |
| NM-12 | 0.234 | 14 | Ultimate (ksi) | 158.4 | 169.1 | 158.6 | | | 125.0 |
| | | | Yield (ksi) | 118.8 | 141.1 | 139.3 | | | 110.8 |
| | | | Elong.(%) | 4.0 | 5.5 | 5.0 | | | 7.0 |
| | | | Hardness ($R_c$) | 32.2 | 36.7 | 33.2 | | | 26.8 |
| NM-19 | 0.251 | 9 | Ultimate (ksi) | 169.2 | 169.4 | Sample cracked | | Sample cracked | |
| | | | Yield (ksi) | 120.0 | 138.8 | in machining. | | in machining. | |
| | | | Elong.(%) | 4.6 | Broke out of ga. | | | | |
| | | | Hardness ($R_c$) | 38.0 | 33.8 | 35.2 | | 33.0 | |
| NM-17 | 0.259 | 52 | Ultimate (ksi) | 175.5 | 176.7 | 168.1 | | 141.5 | |
| | | | Yield (ksi) | 124.9 | 116.9 | 131.2 | | 119.8 | |
| | | | Elong.(%) | 7.0 | 6.5 | 8.5 | | 8.5 | |
| | | | Hardness ($R_c$) | 34.5 | 34.7 | 33.2 | | 27.2 | |
| NM-16 | 0.488 | 15 | Ultimate (ksi) | Sample | 166.6 | 198.5 | | 171.2 | |
| | | | Yield (ksi) | cracked in | 132.6 | 170.9 | | 148.6 | |
| | | | Elong.(%) | machining. | 7.0 | Broke out of ga. | | 6.0 | |
| | | | Hardness ($R_c$) | 39.2 | 36.7 | 40.2 | | 37.5 | |
| NM-15 | 0.660 | 25 | Ultimate (ksi) | Sample | 207.5 | 182.8 | | 191.0 | |
| | | | Yield (ksi) | cracked in | 171.1 | 156.6 | | 168.3 | |
| | | | Elong.(%) | machining. | 4.0 | 5.0 | | 5.0 | |
| | | | Hardness ($R_c$) | 41.5 | 43.2 | 38.7 | | 41.3 | |

* 1 inch gauge length; all other values are for elongation in a 2 inch gauge length.

TABLE II - MECHANICAL PROPERTY/HARDNESS DATA FOR ELECTROFORMED NICKEL-COBALT-MANGANESE ALLOYS

| Sample Number | Alloy Composition Cobalt (Wt.%) | Mn (Wt.%) | S (PPM) | Property Tested | As Deposited | Heat Treated 600°F(24 Hr) | Heat Treated 600°F(48 Hr) | Heat Treated 800°F(4 Hr) | Heat Treated 1000°F(4 Hr) |
|---|---|---|---|---|---|---|---|---|---|
| NCM-28 | 21.2 | 0.057 | 5 | Ultimate (ksi) | 201.7 | 152.0 | 170.2 | 123.2 | 86.7 |
| | | | | Yield (ksi) | 144.1 | 127.2 | 139.4 | 115.2 | 76.2 |
| | | | | Elong.(%) | 4.0 | 6.0 | 4.5 | 11.0 | 29.0 |
| | | | | Hardness ($R_c$) | 44.2 | 39.5 | 37.5 | 25.3 | Below C Scale |
| NCM-27 | 23.0 | 0.067 | 19 | Ultimate (ksi) | 189.1 | 182.3 | 169.6 | 138.1 | 90.1 |
| | | | | Yield (ksi) | 132.7 | 161.0 | 135.1 | 131.8 | 83.3 |
| | | | | Elong.(%) | 6.0 | 3.5 | 4.5 | 9.0 | 26.0 |
| | | | | Hardness ($R_c$) | 39.8 | 38.3 | 38.2 | 35.0 | 20.3 |
| NCM-26 | 26.0 | 0.206 | 22 | Ultimate (ksi) | 244.2 | 252.8 | | 220.2 | 171.0 |
| | | | | Yield (ksi) | 179.5 | 210.2 | | 193.4 | 162.9 |
| | | | | Elong.(%) | 1.5 | 3.0 | | 3.5 | 4.0 |
| | | | | Hardness ($R_c$) | 46.8 | 48.5 | | 46.7 | 37.8 |
| NCM-24 | 32.8 | 0.160 | 21 | Ultimate (ksi) | 242.2 | 243.6 | | 225.2 | 163.4 |
| | | | | Yield (ksi) | 177.1 | 200.1 | | 189.8 | 153.5 |
| | | | | Elong.(%) | 3.0 | 4.0 | | 3.5 | 4.0 |
| | | | | Hardness ($R_c$) | 46.7 | 46.5 | | 44.2 | 35.5 |
| NCM-35 | 37.2 | 0.043 | 45 | Ultimate (ksi) | 209.0 | 177.2 | 185.7 | 135.8 | |
| | | | | Yield (ksi) | 142.3 | 139.9 | 150.9 | 127.5 | |
| | | | | Elong.(%) | 4.0 | 5.0 | 5.0 | 8.0 | |
| | | | | Hardness ($R_c$) | 42.5 | 38.8 | 40.0 | 31.2 | |
| NCM-23 | 37.8 | 0.215 | 44 | Ultimate (ksi) | Sample | 278.1 | | 260.6 | 215.7 |
| | | | | Yield (ksi) | cracked in | --- | | 230.5 | 203.2 |
| | | | | Elong.(%) | machining. | 3.0 | | 3.0 | 3.0 |
| | | | | Hardness ($R_c$) | 50.0 | 50.0 | | 47.3 | 43.0 |
| NCM-34 | 41.2 | 0.041 | 21 | Ultimate (ksi) | 213.9 | 182.1 | 191.9 | 140.3 | |
| | | | | Yield (ksi) | 143.1 | 147.9 | 152.7 | 125.5 | |
| | | | | Elong.(%) | 6.0 | 4.0 | 5.5 | 9.0 | |
| | | | | Hardness ($R_c$) | 43.8 | 38.8 | 40.3 | 31.8 | |
| NCM-40 | 52.5 | 0.043 | 10 | Ultimate (ksi) | 221.0 | 202.6 | 197.2 | 169.9 | |
| | | | | Yield (ksi) | 144.9 | 149.9 | 149.6 | 147.2 | |
| | | | | Elong.(%) | 4.4 | 5.0 | 5.0 | 5.4 | |
| | | | | Hardness ($R_c$) | 41.7 | 40.5 | 40.0 | 36.2 | |
| NCM-39 | 54.3 | 0.020 | 6 | Ultimate (ksi) | 203.8 | 186.4 | 181.4 | 136.0 | |
| | | | | Yield (ksi) | 128.5 | 138.4 | 132.3 | 121.5 | |
| | | | | Elong.(%) | 3.7 | 6.0 | 7.0 | 10.7 | |
| | | | | Hardness ($R_c$) | 39.7 | 38.5 | 38.0 | 35.0 | |
| NCM-41 | 57.4 | 0.018 | 20 | Ultimate (ksi) | 198.7 | 180.9 | 177.7 | 134.1 | |
| | | | | Yield (ksi) | 132.5 | 123.1 | 126.1 | 111.3 | |
| | | | | Elong.(%) | 4.3 | 8.5 | 10.5 | 9.5 | |
| | | | | Hardness ($R_c$) | 40.2 | 37.3 | 36.5 | 30.2 | |

electroformed alloys appears superior to that for nickel-manganese alloy. Greater hardness also appears to be achieved with the nickel-cobalt-manganese deposits. Although manganese contents are not great when compared to nickel-manganese alloys, the relative affect on mechanical properties is unexpectedly high. Stress remains a greater problem with the nickel-cobalt-manganese alloys than with nickel-manganese specimens.

The current nineteen sets of Ni-Mn and Ni-Co-Mn samples will complete heat treatment and testing by mid-September. By the next reporting period we will expect to have a more complete definition of the electrolyte composition and electroforming parameters to be used in producing the round bars for submission to MSFC. A 250 gallon plating bath is currently available for adjustment of manganese to the level desired for the final test bars.

## III. CURRENT PROBLEMS

Slight delays are being experienced in processing test results through metallurgical testing and evaluation due to other work of high priority. This problem now appears rectified and we can expect all backlog of testing to be complete during September.

## IV. WORK PLANNED

1. Evaluate the 343°C (650°F) heat treatment on ductility improvement in comparison with data for 315.6°C (600°F).

2. Determine if the use of stress reducer in combination with high electrolyte manganese content results in superior mechanical properties while providing adequate ductility after moderate heat treatment.

3. Confirm performance of latest sets of specimens showing useful properties by testing at 149°C (300°F) and 260°C (500°F).

4. Tabulate test data for those nickel-manganese specimens and deposition parameters showing best combination of mechanical strength and ductility. Advise MSFC personnel of results for final selections and recommend most optimum parameters for duplication as round test bars.

4. Defer microstructure evaluations and creep testing pending final parameter selections.