25 September 1986

AD-A174 475

Scientific Officer
Program Manager Metallic Materials
Office of Naval Research
800 North Quincy Street
Arlington, Virginia 22217

Attn: Dr. Don E. Polk
Ref: N00014-84-C-0440

Subject: Submittal of the Final Report, FR #19546

Gentlemen:

In accordance with the applicable requirements of the contract, we herewith submit one (1) copy of the subject report.

A DD Form 250 is also enclosed for your acknowledgement. If the subject data is acceptable to your office, we request the Technical Program Monitor's endorsement in Block 21B and Block 22. Please return one copy of the executed DD Form 250 to the attention of the undersigned.

Very truly yours,

UNITED TECHNOLOGIES CORPORATION
Pratt & Whitney
Engineering Division

Margaret B. Hall
Contract Data Coordinator

cc: With enclosures:

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RAPID SOLIDIFICATION AND CONSOLIDATION OF IRON BASE ALLOYS

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September 1986

Final Report For Period
1 September 1984 to 31 July 1986

Prepared for:
Office of Naval Research
600 N. Quincy Street
Arlington, Va 22217
This final report on Contract N00014-84-C-0440 describes work carried out at the Engineering Division of Pratt & Whitney (a division of United Technologies) during the period 1 September 1984 through 31 July 1986. The principle investigator/program manager at P&W was Dr. R.G. Bourdeau who coordinated all work with Dr. G.B. Olson of MIT. Dr. B.A. MacDonald was the ONR scientific officer in charge of the program.
This program produced and consolidated rapidly solidified powders of iron base and steel compositions for the Massachusetts Institute of Technology (M.I.T.) for use on a companion O.R. program. A total of seven compositions were converted to rapidly solidified powder in a P&W experimental rotary atomization device. The powders were screened, canned in extrusion containers, extruded to bar stock and delivered to MIT. Manganese containing TRIP steels and an iron base alloy were produced with refined structures where the contaminants in the steel such as sulfides were finely dispersed. Lanthanum added to the alloy steel gettered the phosphorus and refined phosphides as well as oxysulfides were produced for grain coarsening resistance. The latter steel is being evaluated in sharp-crack $K_{IC}$ toughness and $R_{ISC}$ stress corrosion resistance. In addition, Fe-Ni base compositions were produced which are being used to study the effect rapid solidification in as solidified material.
SECTION I

INTRODUCTION

The program has as its objective the production and consolidation of rapid solidified powder of iron base and steel compositions for the Massachusetts Institute of Technology (MIT). The compositions are for use by MIT on a companion ONR program. An earlier program demonstrated that the contaminants in steels, i.e. sulfides and silicates, could be finely dispersed by rapid solidification and that these were effective in providing resistance to grain coarsening during subsequent treatment at high austenitizing temperatures (ref 1). The current program has produced TRIP steels and an alloy steel with rare earth (lanthanum) additions. The lanthanum was used to scavenge phosphorus for grain coarsening resistance as well as property improvements. In addition, Fe-Ni alloy powders were produced for solidification studies.

A total of seven compositions (four alloy steel and three Fe-Ni compositions) were converted to rapidly solidified powder. The powders were screened, vacuum canned, extruded to bar stock and delivered to MIT for use on their program.
SECTION II

PROGRAM PLAN AND SCHEDULE

The objective of this technical effort was to produce a number of iron base alloy and steel compositions for the Massachusetts Institute of Technology (MIT). The program was to be performed in full cooperation with MIT which is conducting a basic program to demonstrate that the use of rapid solidification will produce superior fracture toughness and stress corrosion properties in high strength steels. The program was to produce also a number of Fe-Ni base alloy compositions for use in basic research on solidification.

The program plan and schedule consisted of two tasks to be carried out simultaneously. A task to produce the rapidly solidified powder from cast ingot obtained from a steel producer, and a task to screen and consolidate the powders by extrusion in a P&H extrusion press at conditions determined in cooperation with MIT.

Due to delays in assigning an MIT graduate student to the program and due to delays in obtaining alloy melt stock from a steel supplier, the program schedule was extended from 12 months to 23 months. This delay was accomplished without reduction in the level of effort.
ALLOY PROCESSING

The iron base alloy and steel compositions provided as cast billets by the LTV Steel Corporation were atomized to rapidly solidified powder in one of two small scale experimental P&W atomization devices. Some of the alloy powders were consolidated to bar stock by extrusion and all material was shipped to the Massachusetts Institute of Technology (MIT), for use in their studies dealing with the effects of rapid solidification on alloy steel structure and properties. This report covers the processing and consolidation of the rapidly solidified powder compositions produced during the program.

Powder Processing

The base compositions processed were in the form of cast billets approximately 4 inch in diameter by 7 inch long each weighing about 25 lbs. The billets were remelted in the P&W atomization apparatus and converted to powder.

The rapidly solidified powder was produced in an experimental powder apparatus (ACT 500000) described in a prior report (ref 3). The liquid metal was vacuum induction remelted in a stoppered Al₂O₃ crucible and the liquid metal metered through an Al₂O₃ nozzle onto a rotating atomization disk. The liquid metal superheated some 300°F (150°C) was poured in a helium atmosphere at an average rate of 20 to 30 lbs per minute (150 to 225 gms per second) onto the disk rotating at a speed of 24000 RPM. The liquid metal desintegrated at the edge of the disk into droplets and these were ejected at a velocity of about 152 meters per second into the helium atmosphere. Droplets solidification rate were estimated to be in the range of $10^5$ to $10^6$ degrees per second (ref. 2). The alloy compositions processed and the powder yields obtained are presented in table 1.

Powder size distributions for the alloys produced were found to be typical of iron base alloys and similar to the powder distributions produced on contract NCC014-B1C-0016 (ref 3). The average powder particle diameter was approximately 70 microns.
### Table 1 Powder (-80 mesh) Produced for MIT

<table>
<thead>
<tr>
<th>Billet</th>
<th>Alloy Composition</th>
<th>XSR #</th>
<th>Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>V-153</td>
<td>Fe-4CNi</td>
<td>II-423</td>
<td>3300 gms</td>
</tr>
<tr>
<td>V-188</td>
<td>Fe-30Ni</td>
<td>II-430</td>
<td>2070 gms</td>
</tr>
<tr>
<td>V-187</td>
<td>Fe-20Ni</td>
<td>II-431</td>
<td>5180 gms</td>
</tr>
<tr>
<td>V-666</td>
<td>Fe-10Ni-16Cr-3.66Mn</td>
<td>II-424</td>
<td>4600 gms</td>
</tr>
<tr>
<td>V-149</td>
<td>Fe-10Ni-16Cr-0.4Ti</td>
<td>II-425</td>
<td>4380 gms</td>
</tr>
<tr>
<td>V-150</td>
<td>&quot;</td>
<td>II-426</td>
<td>6080 gms</td>
</tr>
<tr>
<td>V196-1</td>
<td>Fe-2Ni-1.5Ti-0.4C-0.15La</td>
<td>II-428</td>
<td>1895 gms</td>
</tr>
<tr>
<td>V196-2</td>
<td>&quot;</td>
<td>II-429</td>
<td>1210 gms</td>
</tr>
<tr>
<td>V196-1</td>
<td>&quot;</td>
<td>II-432</td>
<td>2050 gms</td>
</tr>
</tbody>
</table>

Processing of the last two billets (V196-1 and V196-2) caused difficulties due to blockage of the ceramic nozzles (used to control the flow of liquid metal) soon after the start of atomization. In process runs II-428 and II-429, the liquid metal flow ceased 33 seconds and 52 seconds respectively after the start of processing. Energy dispersive X-ray microanalysis of the cross sections of the ceramic nozzles indicated the presence of lanthanum compounds at the nozzle-metal interface but none could be observed in the bulk of the solidified metal in the throat of the nozzle. The blockage during the last three process runs was unusual and therefore it appeared that the lanthanum contributed to the problem. The last process run II-432 which was a re-run of the remaining V196-1 billet produced similar results. The powder yields for the latter three process runs were less than half the expected yields but sufficient material was produced for MIT to conduct its research studies.

### Powder Consolidation

The iron base alloy powders produced were all sieved through an 80 mesh (177 micron) screen in a high purity helium atmosphere containing less than 3 ppm of oxygen. The first three powder alloys in Table 1 were screened and shipped to MIT for use in their studies dealing with solidification rate effects. The remainder of the alloys in Table 1 were consolidated by extrusion prior to shipment to MIT.

The canning of powders for consolidation by extrusion was accomplished in a vacuum of at least $10^{-5}$ torr by passing the powder through the hot dynamic outasser whose schematic is shown in Figure 1. The stainless steel containers 2.9 inch diameter by 7 inch long with 1/2 inch diameter inlet filler tubes (schematic shown in Figure 2) were filled with a predetermined volume of powder weighing about six (6) pounds. For the last three smaller powder lots, II-428, II-429 and II-432, smaller extrusion cans with a 1.75 inch internal diameter were employed. Sealing of the containers was accomplished by pressure...
Figure 1  Schematic of Hot Dynamic Outgasser.
Figure 2 Extrusion Can

- Crimp
- Nickel 200
- 347 Stainless Steel
- 7"
- 0.2"
- 2.9"
welding the inlet tubes in a hydraulic press followed by TIG welding to ensure a permanent seal.

Powders from process runs II-424, II-425 and II-426 were extruded at a ratio of 25 to 1 while the remaining powders from II-428, II-429 and II-432 were extruded at a ratio of 20 to 1. The latter powders were all extruded at a temperature of 2000°F (1093°C). In addition, a mild steel can filled with -27C mesh 434C steel powder which had been produced on prior contract RCCC14-£1-C-3016 was extruded at a ratio of 20 to 1 at 2000°F (1093°C). All extrusions were satisfactory requiring no additional processing prior to their evaluation. These extrusions were submitted to MIT for use on their program.

Summary of Results

The rapidly solidified powders produced for MIT were either screened through a -80 mesh screen, as for the three Fe-Ni alloy compositions, or canned and extruded as for the remaining steel compositions. The ThIF steel compositions (II-424, II-425 and II-426) were found to have finely dispersed oxysulfides and silicates while the steel with lanthanum additions (II-428, II-429 and II-432) was found to have a fine distribution of phosphides in addition to oxysulfides. The latter steels are undergoing further study at MIT to evaluate K_{CC} stress corrosion properties. A substantial increase in stress corrosion resistance over conventionally produced material is anticipated for the rapidly solidified material. The investigation of solidification rate effects in the Fe-Ni alloys (II-423, II-430 and II-431) is in progress.
REFERENCES


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