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APPLICATION OF SUPERPLASTIC STEELS

E. R. Slaughter, R. G. Bourdeau Pratt & Whitney Aircraft Group Government Products Division Box 2691, West Palm Beach, Florida 33402

JUNE 1978

Semiannual Report

Period 1 December 1977 Through 31 May 1977

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SUMMARY

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Thermomechanical processing of cast ultrahigh-carbon steel ingots, to produce a fine twophase structure required for superplastic behavior, is presently effective for rolled stock and 2- to 3-**ib** forgings. For larger billets, ultrahigh-carbon steel powders and an iron-boron alloy powder can be hot compacted at relatively low temperatures to produce the desired fine grain structures. The powder base ultrahigh-carbon steel was forged to a difficult-to-forge shape which normally can be produced only of materials with fine superplastic grain structures.

With a laser rapid melting-rapid solidification method, it was found that an Fe-Ti-B alloy with a Ti/B ratio greater than 0.5 could be produced with a fine (200 to 2000 Å) TiB₂ dispersion. Rapidly solidified powders of ferrous alloys containing Ti and B in the ratio of TiB₂ were found to contain similar fine dispersions which appeared to be effective in inhibiting grain growth. An alloy whose composition was AISI 4340 steel plus TiB₂ was hardened by the martensitic transformation. Preliminary results indicate that a hardenable alloy steel containing a dispersion of TiB₂ may be superplastic.

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SECTION I

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INTRODUCTION

Superplasticity in metal alloys permits forming to complex shapes and at relatively low stress levels. In some cases, as with nickel-base superalloys, superplasticity makes it possible to forge to complex shapes a material which is considered unforgeable. In other cases, as with a Zn-Al alloy, forming operations used for plastics are employed to produce intricate parts to close tolerances and with faithfully reproduced surface textures. For steels made superplastic, economics from processing and subsequent machining should be attainable, and it can be expected that gains in properties associated with a very fine grain size may also be in the offing.

Research conducted at Stanford University by O. Sherby et al \bullet has shown that superplasticity can be achieved in simple but ultrahigh-carbon steels (1.3 to 1.9 weight⁽ⁱ⁾ C) by thermomechanical processing in such a way that a very fine grain size in the micron range is produced. The very high carbon contents are required to produce sufficiently high concentrations of cementite (20 to 29 volume⁽ⁱ⁾ Fe₃C) to stabilize the ferrite at working and superplastic deformation temperatures. By working in the austenite-cementite range, the massive cementite phase present in the original casting is broken up and spheroidized. And by further working in the ferrite-cementite range, the transformed austenite or pearlite is also spheroidized, while the structure is refined to a very small grain size. Various thermomechanical methods were employed to produce grain refinement but in all cases the final structure, for superplastic behavior, was an equiaxed ferrite grain in the order of 1 to 3 microns stabilized with a submicron spheroidized cementite precipitate.

A material can be defined as superplastic if the strain rate sensitivity exponent, m. in the relation $\sigma = K \epsilon^m (\sigma \text{ is the flow stress, } \epsilon \text{ is the strain rate and K is a material constant)}$ approaches 0.5 and high elongations (to 500%) are achieved. According to Stanford University researchers, the ultrahigh-carbon steels are superplastic at warm temperatures (below the γ to α + Fe_sC transformation temperature) at a strain rate in the order of 1% per minute.

The purpose of this Advanced Research Project Agency (ARPA) sponsored program is to apply the principles developed at Stanford University to the precision forging of steel shapes by the GATORIZINGTM forging process, and to determine the benefits to be realized by the use of superplasticity in steels. Three parts, which are believed to be representative of mass-produced items, were selected to evaluate the forging of superplastic steels. These are: (1) an aft closure of a laser guided missile, (2) a forward control section of the same missile, and (3) a die assembly for hot forging pinion gears.

The program is a 27-month effort starting with an evaluation of the forgeability of an ultrahigh-carbon steel composition produced initially as *a* cast billet. Alternately, the same alloy is to be produced as a powder to determine the advantages gained by the elimination of massive cementite. In conjunction with the above tasks is an evaluation of the potential of alloying to alter mechanical properties without loss of superplasticity. Finally, subsequent to a precision forge evaluation and component fabrication, the economic and material benefits derived from superplasticity in steel will be assessed.

This is the second semiannual technical report covering the period from 1 December 1977 through 31 May 1978. It deals with the thermomechanical processing of cast ingots of ultrahighcarbon steel and with the consolidation of powders of the same composition to produce a fine duplex microstructure characteristic of superplastic materials. It deals also with the alloy development phase of the program.

^{*} Sherby, Oleg D. et al. 'A Summary Report on Superplastic Ultrahigh-Carbon Steels.'' Advanced Research Project Agency, Grant No. DAHC-15-73-G15, February 1977

SECTION II

PROCESS DEVELOPMENT

FORGING

Cast billets of 1.5% carbon steel were forged above and below the austenite to pearlite transformation temperature to produce the fine two-phase microstructure which was developed by rolling by Prof. O. Sherby of Stanford University. This was the first attempt at scale-up performed on 6-in, dia ingots using the procedure described in the first semiannual technical report of this contract. Briefly, the ingots were hammer forged at approximately 1400 to 1500°F to break down the as-cast structure and subsequently reforged at about 1100°F to further refine the structure and spheriodize the cementite. Microscopic examinations of the forged ingots revealed that the desired fine duplex ferrite-cementite structure was produced. Figure 1 shows this base structure of ferrite with spheriodized cementite. However, also present in the structure was a coarse network of proeutectoid-cementite at prior austenite grain boundaries. Figure 2 shows, at a low magnification, the carbide which was not dispersed during the forging operation.

Following this investigation, special tasks were initiated to determine the extent of deformation required as a function of ingot volume to break down the massive cementite which forms during cooldown through the austenite-cementite phase field. First, ingots of different size were forged in a hydraulic press. Then an apparatus was employed to perform torsion tests on samples cooled at a rate typical of an 8-in. dia ingot. The torsion test permitted the deformation parameters to be varied conveniently over wide ranges and required only small samples to simulate the forging of large billets. The following discussion describes the procedures and results obtained.



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Figure 1. As-Forged 1.5% Carbon Steel



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Figure 2. As-Forged 1.5% Carbon Steel

A total of 10 forgings were made in a hydraulic press using ingots which varied in volume from 6 to 64 in? (1 \times 2 \times 3 to 4 \times 4³4 \times 3³s in.). The ingots were forged in steps of 20 to about 50% in two or three directions depending on sample handleability. Some of the smaller ingots were manipulated with a length of steel tubing welded to one face of the ingot for forging in two directions. The larger ingots were manipulated with tongs and forged in all three directions. In general, the ingots were worked continuously to introduce as much work as feasible within the range of available deformation or desired temperature range.

Prior to forging, the ingots were held for 2 hr at 2100°F to completely solution the cementite. The billets were then forged according to one of the following schedules.

- 1. Forge continuously during cooldown from 2100 or 1850°F, which is the start of cementite precipitation, through the γ -Fe₃C phase field.
- 2. Forge from the start of cementite precipitation for approximately 100 to 200°F to create the greatest number of defects in the structure for carbide precipitation.
- 3. Forge at a single temperature of 1750°F in the γ -Fe_sC phase field.

In addition, some of the forged ingots were reforged at 1200 to 1300°F to possibly breakup grain boundary cementite and to spheroidize eutectoid-cementite.

The results were conclusive. Small ingots, i.e., $1 \times 2 \times 3$ in., can be forged to completely disperse the proeutectoid-cementite and to produce a fine two-phase structure similar to that obtained by rolling. Larger ingots approaching the size required for component development contained massive cementite which was continuous in billets forged in two directions but discontinuous in billets forged in all three directions. Figure 3 shows the finer structure produced by forging small ingots, and Figure 4 shows the discontinuous but coarse cementite in the larger ingots.



Figure 3. A [6] int: Insot. Forgeal Through the 5 Fe/C Phase Field



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Figure 1 A 64 in Ingot Forged Through the 7 Fe C Phase Field

The z cond task to investigate the necessary conditions for refining cast structures employed the torsion apparatus shown schematically in Figure 5. An 8-in, dia by 6-in, high superalloy billet, which had a central cavity to accommodate the specimen, gave the apparatus the thermal capacity that simulated an 8-in, dia steel ingot. The steel sample had a gage section 0.437 in, dia by 0.125 in, long.



FD 139276

Figure 5. Hot Torsion Test Assembly

The normal experimental procedure involved heating the apparatus containing a sample to solution the carbon in the sample and then removing the apparatus from the furnace so that it cooled in air at approximately the same rate that an 8-in. dia steel ingot would cool during forging. The sample cooled at nearly the same rate as the inside surface of the apparatus. The sample cooled from 2050°F to the start of cementite precipitation, 1850°F, in 9.5 min. and from that temperature to the transformation temperature, 1265 to 1290°F, in 22 min.

During cooling through the temperature range from 1850°F to the transformation temperature, the specimen was deformed by applying a torque manually. In a series of experiments, the deformation was applied in some instances by torque in one direction and, in other cases, the sense of the torque was changed after rotations of 20 to 30 deg. To simulate a forging operation, the strain was applied in increments with intervening intervals of no strain. The strain at the surface of the gage section varied from 0.1 to about 1.0 per increment and the interval from 5 sec to 2 min.

In the series of experiments, the total strain was generally increased and the interval between deformation increments decreased in attempts to eliminate coarse grain boundary cementite. A typical structure is shown in Figure 6. For the final experiment in this series, an interval between deformation increments of 5 sec and a total strain on the order of 20 was employed. The grain boundary cementite was not broken up and is as shown in Figure 6.

Finally, an experiment was performed in which only the specimen was heated. It was transferred to the apparatus, which was at room temperature. The sample cooled from 1850°F to the transformation temperature in 125 sec and the specimen was strained in increments of about 0.1 at 2-sec intervals, yielding a total strain of about 6. The distribution of the proeuctectoid-cementite closely approached that obtained by rolling or forging small samples.

From the previous discussion, it was concluded that the morphology of proeutectoidcementite is affected by strain, cooling rate, and possibly by strain rate during cooling through the carbide precipitation range. The amount of strain required to disperse the cementite increased with decreasing cooling rate or increasing billet size.



Mag: 100X FD 139277 Figure 6. Torsion Sample - 1.5% Carbon Steel

POWDER PROCESSING

The use of powder as the starting material is an alternate approach for producing fine grain. ultrahigh-carbon steel. It was shown in the preceding section that the slow cooling of the ultrahigh-carbon steel through the austenite plus cementite temperature range results in the formation of continuous grain boundary networks of cementite which can only be dispersed by thermomechanical processing. These networks can be avoided by rapid solidification, and this effect in powders can be retained through consolidation and fabrication of the material into components.

The alloy development phase of this program, which involves boron-containing ferrous alloys, depends upon the rapid solidification inherent in powders. Therefore, the processing of a prototype alloy, binary iron-1^c boron alloy, was also studied not only to learn how to process boron-containing alloys but also to confirm that the laser welding technique used as a screening test for alloy development was valid.

Argon atomized powders of the ultrahigh-carbon steel and the iron-boron alloy were obtained from a commercial source. The nominal composition of the carbon steel was:

carbon	$1.5^{c}\epsilon$
manganese	1.0°č
vanadium	0.15°č
silicon	0.1% maximum
iron	balance

6

Both previously discussed alloys were compacted by hot pressing at 1225 F to prevent the formation of austenite and 20,000 psi pressure. The ultrahigh carbon steel appeared to be completely dense metallographically and the structure consisted of ferrite plus fine spheriodized cementite. The boron alloy was on the order of 99 – dense. Some of the boron alloy powder particles retained their original shape and dendritic structure, but most had deformed and recrystallized into a fine grained structure. The ultrahigh carbon steel was judged to be suitable for final torging as compacted, while it was felt that the boron alloy needed a prior forging operation to fully recrystallize its structure.

Since it is known that time powder solidities more rapidly than coarse powder, two size tractions (-80 + 140 mesh) and -270 mesh) were processed to study the effect of solidification rate upon structure, dispersed phase particle size and mechanical properties of the boron alloy.

Billets of the high-carbon steel and both mesh fractions of the boron allov were hor isostatically pressed (HIP) at 1325 F. Also, the boron allov billets were forged at 1550 F to provide material for microstructural and mechanical property evaluation. In addition, an ultrahigh carbon steel billet was upset to a strain of 2.0 at 1525 F and a strain rate of 0.01 min to determine the effect of slow strain rate deformation upon material properties.

The structure of the HIP steel consisted of ferrite and partially spheriodized-cementite. The size of the ferrite grains and the cementite particles varied from region to region. The size of the regions of uniform grain and particle size corresponded more or less to the size of powder particles. Apparently, the size of the ferrite grains varied from powder particle to powder particle, but powder particle boundaries could not be discerned in the structure after consolidation. Typical and coarse structures are shown in Figures 7 and 8.



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Figure 7. Typical Structure of HIP Steel



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Figure 8. Coarse Structure of HIP Steel

The ferrite grain size and cementite particle size were larger in the steel upset at 0.01/min while the ferrite grain size increased by roughly a factor of 5 as shown in Figure 9.

The structure of the boron alloy consisted of ferrite and a dispersed boride phase. A typical structure is shown in Figure 10. The structures at ten randomly selected areas in forging of the fine and the coarse powder fractions were compared. The average structure of the fine powder forging was finer than that of the forging of the coarse powder fraction, but the variation in grain and particle size from region to region within a forging was larger than the difference between the means of the materials.

The structure of the forgings resembled that of the laser welded samples of the same composition to the extent that the laser weld technique is considered adequate as a screening test capable of detecting large effects of composition on grain size and dispersed phase particle size.

The significance of the foregoing results is that fine grain structures can be obtained by consolidating high-carbon steel and iron 1% boron powders. Also, duplex structures containing a spheriodized and stabilizing second phase can be obtained directly.

The room temperature mechanical properties of steel and the boron alloy are given in Table 1.



Figure 9 Structure of Steel Upset at 0.011 Mm



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Figure 10. Typical Structure of Iron-Boron Alloy

Alloy	Condition	UTS (ksi)	YS (ksi)	Elongation (*_)	Reduction in Area (*)
Ultrahigh-carbon Steel	НГР	120,3	110.1	6.7	6,5
	Slow strain rate				
Terre 11 Barrie	forging	91.0	88,8	12.7	14.4
Iron-1% boron	Forging Coarse powder	80.8	68.9	24.0	48.3
	Forging	(4),0	90,9	24.0	40.4
	Fine powder	87.0	79,3	24.0	44.3

TABLE 1. ROOM TEMPERATURE MECHANICAL PROP-ERTIES

The ultimate tensile strength and the yield strength of the ultrahigh-carbon steel as HIP were lower the strengths reported by Prof. O. Sherby for this steel rolled extensively in the austenite plus cementite range and the ferrite plus cementite range. The lower strengths obtained on this program were associated with the lack of extensive warm work. The grain and cementite particle size growth during the slow strain rate forging operation was the apparent cause of the decrease in strength during that operation.

The strength of the iron-boron alloy from the fine mesh fraction powder was higher than that from the coarse powder and apparently reflected the finer grain size of the former.

A slab of the ultrahigh-carbon steel 0.4-in. thick was austenitized at 1400°F for 1 hr and water quenched to determine its hardenability. A hardness traverse on a section indicated that the maximum hardness, VPN 851, extended only to a depth of 0.1 in. The hardness at the center was VPN 402.

The behavior of the ultrahigh-carbon steel and the iron-boron alloy during the GATORIZ-INGTM (superplastic forging) process was studied as a function of temperature. Billets 1-in. dia by 0.625-in. high were lubricated with boron-nitride and isothermally forged at a pressure of 30,000 psi for 30 min. The die, which was available and produced a shape that was difficult to forge, produced an airfoil extrusion that was both tapered and twisted. Due to the twist and taper, the resistance to extrusion increased with the length of the airfoil. Therefore, airfoil length was a measure of the merit of an alloy for GATORIZING. One sample of a commercial alloy steel, AISI 4140, was included in the study for comparison.

The lengths of the airfoils are given in Table 2. Typical airfoils are shown in Figure 11. The lengths of the airfoils can generally be rationalized on the basis of temperature, grain size and the allotropic form of the alloy. The decrease in flow stress with increasing temperature prevailed for the ultrahigh-carbon steel. The airfoil length increased with temperature despite the allotropic change between 1300 and 1550°F which increased the strength of the steel. The grain size of the steel during GATORIZING could only be inferred from the transformed structure in the forging, but it appeared that little growth occurred at 1550°F. Using the 1700°F forging, the proeutectoid-carbides and the grain size had grown, but the proeutectoid-carbides still appeared to be randomly distributed. At 1850°F the carbides and the grain size had grown further and many of the proeutectoid-carbide particles appeared to be in the austenite grain boundries. However, unlike the alloy produced from an ingot, the grain boundry carbides did not form a continuous network.

The airfoil lengths of iron-boron alloy derived from coarse and fine powders cannot be rationalized on this basis. The grain size of the material from fine power was finer than that of the coarse powder material at 1300°F and the airfoils from the coarse powder were longer but their grain sizes were the same at higher temperatures. The iron-boron alloy formed a longer airfoil at

1550 F than the ultrahigh-carbon steel because it was territic and the carbon steel austenitic, despite its larger grain size. The grain size of the boron alloy at 1700 F was over 50 microns and the grain size effect predominated over the temperature effect when compared to the 1550 F forging.

TABLE 2. EVALUATION OF ULTRAHIGH-CARBON STEEL AND IRON-BORON ALLOY FOR GATORIZING**

Arte : Long to					
- GATORIZING Teggenstere - F	I manual Carbon			NSE : :	
N I	0.249	0.309	A 1 1 1 1 1 1 1 1 1	•	
· * * •	N 877	0.1.26.000	11 m h	10 j. a	
, n :	2.240	0.64640	100 B (172		
, * 1+1	1.40				



Figure 11. Airfoil Forgings

The ultrahigh-carbon steel was GATORIZED* at 1800 F into a near net shape one-third scale turbine disk. The forging demonstrated the ability of the material to fill the thin integral axial spacer extending from the rim of the disk, Figure 12.



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Figure 12. Subscale Turbine Disk Forging

ALLOY DEVELOPMENT

During this report period, screening of numerous alloys using more shallow laser welds on small arc cast button ingots was continued.

Iron-Carbon-Boron Alloys

In these alloys the dispersed phase was coarse lamellar. The boron appeared to prevent the solution of the carbon which is required for strengthening by the martensite reaction. This system appears unlikely to provide useful superplastic alloys. The addition of 2.5% aluminum and 1.5% silicon to iron-boron alloys as solid solution strengtheners and ferrite stabilizers enhanced the effect of the boron in reducing grain size. Fine grains were produced with only 0.32% boron, but more than 0.5% boron was required in the binary iron-boron alloys to produce the same grain size.

Iron-Titanium-Boron Alloys

The boron content in these alloys was varied from 0.23 to 0.56% by weight. The atomic ratio of titanium to boron in these alloys varied from 0.09 to 1.13. For all boron contents, the morphology of the dispersed phase(s) varied after the standard thermomechanical process with the titanium to boron (Ti/B) ratio. For Ti/B ratios less than 0.5, the dispersed phase resembled that found in the binary iron-boron alloys. For Ti/B ratios of approximately 0.5 or more, the dispersed phase was approximately one order of magnitude finer as shown in Figures 13 and 14. The nominal boron contents and the titanium ratios are given in Figure 15.

According to Zener*, the limiting grain size of an alloy is proportional to the radius of inclusions and inversely proportional to the volume fraction of the inclusions. He assumed uniform spherical inclusions randomly distributed; thus, his relationship only approximates actual cases. According to this relationship, the iron-titanium-boron alloys promise to attain the fine grain structure necessary for superplasticity with greatly reduced volume fraction of the dispersed phase. Reducing the volume fraction of the dispersed phase would not only reduce the cost of alloys, but lessen the effects of the brittle dispersed phase on room temperature ductility and toughness. To verify these potential advantages, an alloy was produced as rapidly solidified powder.

^{*} Zener, C., Private Communication to C. S. Smith cited in Trans. AIME, Vol 175, pp. 15-51, 1949.



Figure 13. Typical Fe-Ti-B Alloy With Ti/B Less Than 0.5



Figure 14. Typical Fe-Ti-B Alloy With Ti/B Greater Than 0.5





FD 139286

Figure 15. Iron-Titanium-Boron Alloys

A 50-1b melt of the alloy was converted to powder in an experimental apparatus that achieves cooling rates in excess of 10° °C/sec, by means of a helium quench, for powder particles of -140 mesh. The alloy's nominal composition was iron, 1.5% aluminum, 1.33% titanium, 0.6% boron by weight, and the atomic ratio of titanium to boron was 0.5. Since this powder was produced recently, the evaluation has been limited to consolidation studies.

The alloy compacted by hot pressing at $1725^{\circ}F$ for 1 hr appeared to have two dispersed phases. One was very fine and nearly spherical resembling the dispersed phase in the screening tests of the iron-titanium-boron alloys. The other phase was more voluminous and the particle size was considerably larger, as shown in Figure 16. The alloy compacted at $1550^{\circ}F$ by hot pressing and then forged after annealing for 2 hr at $1800^{\circ}F$ had only the finer second-phase particles, as shown in Figure 17. It was postulated that at the lower temperatures the boron is associated with Fe₂B while the titanium is dissolved in the ferrite phase. At high temperatures the Fe₂B dissociates and the boron dissolves in the ferrite where it reacts with titanium to form the stable TiB₂ phase.

The dispersed phase particles were mostly less than 1000 Å in diameter, with particles as small as 200 Å as shown in Figure 17. Note that the extracted particles, which appear black, had a similar size distribution to the replicated particles, which appear light. Transmission microscopy of a single sample tended to confirm the size distribution as revealed by the replica technique.



Figure 16. Fe-Al-Ti-B Alloy Hot Pressed at 1725°F



Figure 17. Fe-Al-Ti-B Alloy Forged at 1800°F

The grain size could not be determined from replica, Figure 17. Transmission microscopy of the material revealed many boundaries, but it could not be determined whether the boundaries were high- or low-angle boundaries. Only high-angle boundaries are believed to contribute to superplasticity.

While the iron-aluminum-titanium-boron alloy promises to be superplastic at high temperature and ductile at room temperature, its room temperature strength would be derived from a solute strengthened ferrite, a fine grain size, and a low volume fraction of the dispersed phase. Figure 18 shows a strip bent at room temperature. Since the iron-boron binary alloy had a yield strength of 79 ksi with no solute strengthening and, presumably, a coarser grain size than the Fe-Al-Ti-B, it is not unreasonable to expect a yield strength of 100 to 150 ksi from this type of alloy. This combination of mechanical properties may be well suited for some applications, but this program has as a goal the production of a missile component requiring higher strength material.

The Fe-Al-Ti-B alloy is regarded as a promising material, but most importantly for this program, a logical step in the development of a higher strength alloy. It can be viewed as a relatively simple system combining a TiB_2 dispersion in a stable ferrite matrix. A major part of the effort in the next report period will be spent studying the effects of thermomechanical processing parameters upon the TiB_2 dispersion and the grain size.

A high strength and superplastic alloy steel may be obtained by superimposing a TiB_2 dispersion on a conventional high strength alloy steel. Toward this goal, an alloy whose composition was that of 4340 steel plus 1.26% Ti and 0.56% B was converted to rapidly solidified powder. This material has thus far been studied for compaction characteristics, but one consolidated sample was produced whose structure, as quenched from 1600°F , apparently consisted of a fine dispersed phase, martensite, and a small amount of an unidentified phase as shown in Figure 19. The as-forged hardness of about DPH 320 was increased to DPH 526 by quenching from 1600°F . As discussed previously, boron additions to iron-carbon alloys seemed to inhibit the solution of the carbon in austenite and prevent the martensite transformation. However, the titanium may have combined preferentially with the boron in the modified 4340 alloy since a substantial fraction of the carbon must have been in solution at 1600°F for the martensite transformation to occur on quenching.

The results on the modified 4340 alloy are preliminary. They indicate that there is a possibility that this alloy can be made superplastic and that very fine grained austenite can be made to undergo the martensitic transformation. The room temperature mechanical properties of a martensitic structure transformed from very fine grain austenite is generally expected to be superior to those from normal austenite.



Mag: 3X

FD 109289

Figure 18. Bend Specimen of the Fe-Al-Ti-B Alloy



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Figure 19. Modified 4340 Steel Quenched from 1600°F