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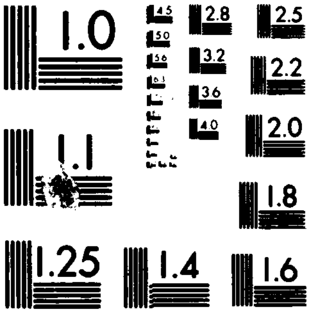
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Rapid solidification of a high-phosphorus austenitic steel produces a fine cellular solidification structure containing an amorphous phase at the cell walls. The amorphous phase, which is stable to $\sim$500° C, is enriched in phosphorus and chromium, but contains significantly less phosphorus than conventional glass-forming alloys. Hot consolidation of powders produces a chemically-uniform metastable austenite which can be effectively precipitation hardened by phospho-carbides.

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ANALYSIS OF A RAPIDLY SOLIDIFIED HIGH-PHOSPHORUS AUSTENITIC STEEL CONTAINING AN AMORPHOUS PHASE

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ABSTRACT

Rapid solidification of a high-phosphorus austenitic steel produces a fine cellular solidification structure containing an amorphous phase at the cell walls. The amorphous phase, which is stable to $\sim$500° C, is enriched in phosphorus and chromium, but contains significantly less phosphorus than conventional glass-forming alloys. Hot consolidation of powders produces a chemically-uniform metastable austenite which can be effectively precipitation hardened by phospho-carbides.

INTRODUCTION

The combined effects of chemical uniformity, extended solid solutitity, and refinement of inclusions permit a greater tolerance for impurities in rapidly solidified materials. Phosphorus, which is a well-known embrittling impurity in high-strength steels, has long been beneficially employed in ferrous powder metallurgy. Phosphorus has also been found to provide effective strengthening of austenitic stainless steels via $(M,P)_{23}C_6$ phospho-carbide precipitation, where normal carbide precipitation is too heterogeneous to allow significant strengthening [1]. This strengthening mechanism may be of particular value for the high-strength metastable austenitic steels known as TRIP steels [2], which achieve high ductility and toughness through a deformation-induced martensitic transformation, but which normally require a difficult thermomechanical treatment to achieve their high strengths. While a precipitation-hardenable TRIP steel would be a desirable alternative. the choice of hardening phases is limited by the requirement for a moderate alloy carbon content ($\sim$0.3 wt.%C) to ensure a high work hardening effect from the deformation-induced martensite. A preliminary study of phospho-carbide strengthened TRIP steel achieved a good combination of austenite strength and transformation behavior, but fracture ductility and fabricability were limited by the severe phosphorus segregation associated with conventional ingot metallurgy [3]. In the first phase of an investigation of the effect of rapid solidification processing on this alloy, some unusual solidification structures were observed, which we report here.

MATERIALS AND PROCESSES

Rapidly solidified powders of an alloy of the composition in Table I were produced by the Pratt and Whitney centrifugal atomization process. After sizing of the powders, electron-transparent specimens were prepared from composite

TABLE I
Alloy Composition

	Fe	Cr	Ni	Si	P	C
wt.%	72.7	16.1	10.3	0.15	0.42	0.30
at.%	71.3	17.0	9.6	0.29	0.74	1.37

foils of powder in electrodeposited nickel by a combination of jet electropolishing and ion-beam milling. Specimens were observed in a Vacuum Generators HB-5 scanning transmission electron microscope (STEM) permitting fine-scale x-ray fluorescence microanalysis.

SOLIDIFICATION STRUCTURES

As observed in a parallel study of a high-sulfur austenitic steel [4], both fcc and bcc primary solidification phases are formed during rapid solidification of this alloy. The bcc, which accounts for about 5% by weight of the -140 mesh (≤120 μm dia.) powder, is found predominantly in the smallest (≤70 μm dia.) particles. An analysis of the thermodynamics and kinetics of solidification for both the high sulfur and high phosphorus alloys suggests that the bcc solidification product is metastable, but kinetically favored by a higher nucleation frequency at heterogeneous nucleants of low potency which operate at the high undercoolings achieved in the smallest particles [4,5].

Fine cellular solidification microstructures are observed in both the fcc and bcc particles. The microstructure of an fcc cellular particle is shown in Figure 1. Microdiffraction analysis of the interiors of the ∿1 μm cells shows that each cell has the same crystallographic orientation. Microdiffraction analysis of the cell walls, however, shows the characteristic ring pattern of an amorphous (glass) phase. Another fcc cellular microstructure and an associated composition profile are shown in Figure 2. It is clear that chromium and phosphorus are strongly segregated to the cell walls. The composition of the amorphous phase at the cell walls is determined as 49Fe-36Cr-7Ni-8P in at.%. Electron energy loss spectroscopy indicates that the amorphous phase is slightly depleted in carbon relative to the crystalline cell interiors.

Fig. 1. Cellular fcc structure. STEM bright-field image and microdiffraction patterns.

A bcc solidification microstructure is shown in Figure 3. The cellular arrangement of this structure is not as regular as that observed in the fcc structures, but an amorphous phase is again found at the cell walls. As in the fcc, microanalysis reveals that the amorphous phase is enriched in chromium and

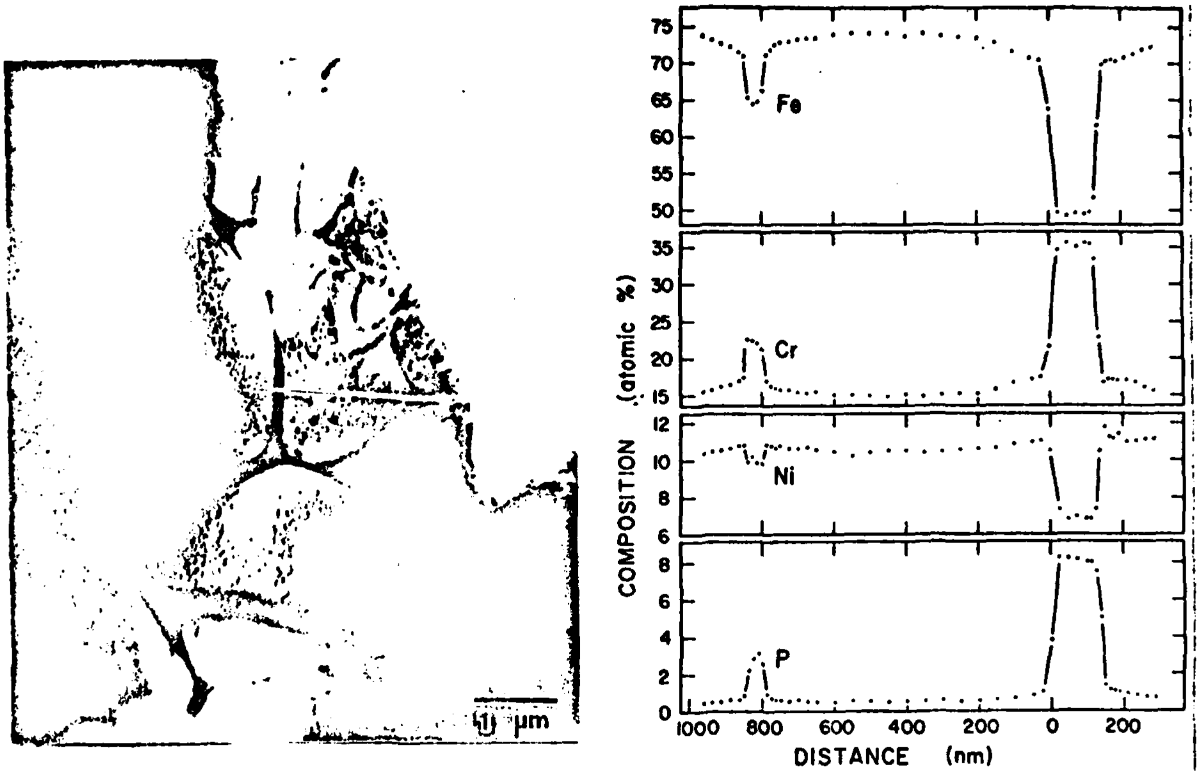

Fig. 2. Cellular fcc structure. STEM bright-field image and composition profile.

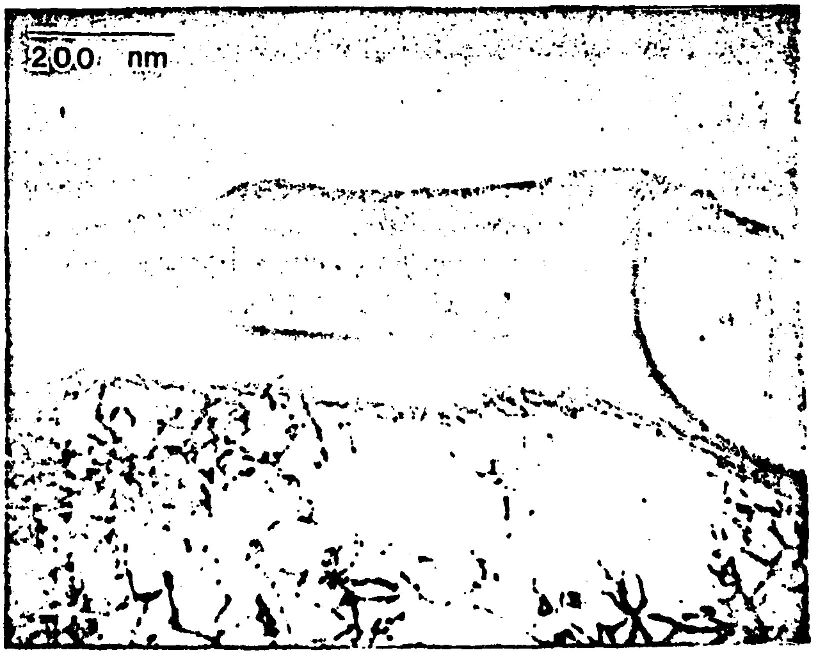

Fig. 3. Cellular bcc structure. STEM bright-field image.

phosphorus.

It is interesting to note that the amorphous phase phosphorus content of $\sim$8 at.% is considerably less than the 15 to 25 at.% metalloid normally required to form metal-metalloid glasses in "bulk" form. If the local conditions leading

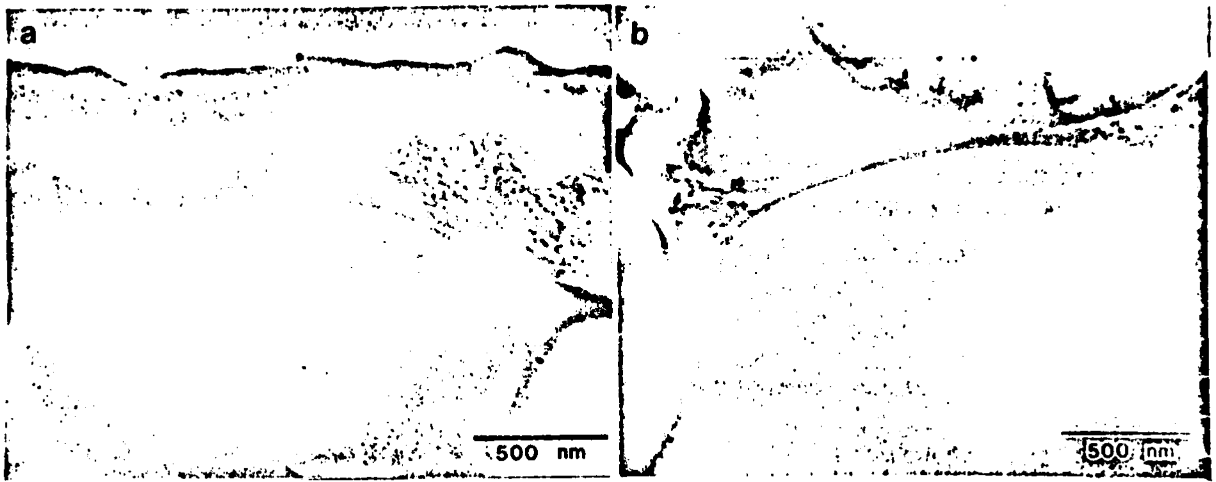

Fig. 4. Cellular fcc structure after 15 minute heat treatment at (a) 480°C and (b) 540°C.

to glass formation in the cell walls can be approximated macroscopically in other rapid quenching techniques, such microanalytical observations may serve as a useful guide to the development of more effective glass-forming compositions. Furthermore, it is evident that the microscopic condition leading to glass formation at the cell walls in these powders is one of suppressed crystal growth rather than nucleation, since the glass is formed in direct contact with the advancing crystal interface. This observation is in line with recent studies emphasizing the importance of crystal growth inhibition in the formation of transition metal-metalloid glasses [6].

Crystallization (devitrification) behavior of the amorphous phase was analyzed in a differential scanning calorimeter. At a thermal scan rate of 10°C per minute, a broad exotherm was detected between 480 and 540°C. Analysis of the exotherm gives a heat of crystallization for the powder sample of 0.285 J/g. Estimating an upper limit of the volume fraction amorphous phase from Figure 1 at ∿2%, this gives a heat of crystallization of the amorphous phase of 14 J/g or 750 J/mole, comparable to heats of crystallization of similar glasses.

Electron micrographs of powder samples heated to 480 and 540°C for 15 minutes are shown in Figure 4. The amorphous phase has crystallized at 540°C but not at 480°C. Microdiffraction shows the devitrified region to be made up of small crystallites on the order of 500 Å and smaller.

The solidification structures of these powders represent an unusual composite material in which the stronger (glass) phase forms a continuous network. While this is normally undesirable for a hard phase which fractures in a brittle manner, metallic glasses are known to fracture by a local shear instability which in this case would be constrained by a cell wall thickness of a few hundred Angstoms. Based on the crystallization measurements, it appears that this unique microstructure could be retained if powder consolidation could be performed below 500°C. It also appears that the metastable bcc structure can be retained up to this temperature, so that magnetic separation of the ferromagnetic bcc powders prior to consolidation would allow both bcc- and fcc-based composites to be examined. The possibilities of powder consolidation by high pressure warm compaction and by dynamic cold compaction are being explored.

HOT CONSOLIDATED MATERIAL

Properties of the alloy after conventional hot consolidation have been examined. Some of the -140 mesh powder was consolidated by 10:1 extrusion at 950°C. This is a sufficiently high temperature to provide complete homogenization of the fine solidification cell structures. Electron microscopy of the as-consolidated material indicated a chemically uniform austenitic structure with a dispersion of chromium-rich carbides.

An austenitizing treatment at 1180°C for 30 min. provided almost complete solution of the carbides and a grain size of ~50 μm. Excessive grain coarsening was found to accompany complete solution of carbides at higher temperatures. This is in contrast to the observed behavior of similarly processed martensitic steels of commercial compositions in which rapid solidification finely disperses stable inclusions which pin grain boundaries at high temperatures [7]. The steel examined here was made from high-purity materials and thus the inclusion effects are apparently absent.

Direct aging of the solution-treated material at 700-750°C gave a grain-boundary initiated cellular precipitation reaction with poor mechanical properties. The cellular reaction could be avoided by cold rolling to a reduction of 45% at 100°C and then aging in the lower temperature range of 600-650°C. A hardness of R_c47 was obtained after 6 hr. aging at 600°C; a comparable hardness level in a conventional TRIP steel would require a warm rolling (400-600°C) reduction in excess of 60%. The tensile properties and transformation toughening behavior of this material are now being evaluated [8].

CONCLUSIONS

Rapidly solidified powders of a high-phosphorus austenitic steel show a fine solidification cell structure in which an amorphous phase is retained at the cell walls. The amorphous phase is enriched in chromium and phosphorus, but contains significantly less phosphorus than conventional glass-forming compositions. The glass crystallizes near 500°C; powder consolidation below this temperature may provide a unique composite material. Hot consolidation at 950°C produces a chemically-uniform metastable austenitic structure which can be effectively strengthened by phospho-carbide precipitation to a hardness of R_c47.

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