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Abstract

Spray forming is an effective way to process many tool steels into near-net-shape molds, dies, and related tooling. The general approach involves depositing atomized droplets onto a refractory pattern in order to image the pattern's features. The pattern is removed and the die insert is mounted in a standard mold base or holding block. This approach results in significant cost and lead-time savings compared to conventional machining. Spray-formed dies perform well in many industrial forming operations, oftentimes exhibiting extended die life compared to conventional dies of the same material and design.

Care must be exercised when spray forming tool steel dies to minimize porosity and control the nature and distribution of phases and residual stresses. Selection of post-deposition heat treatment is important to tailor the die's properties (hardness, strength, impact energy, etc.) for a particular application. This paper examines how the cooling rate during spray processing and heat treatment of H13 tool steel influence phase formation, porosity and residual stress. The performance of spray-formed dies during production runs in forging, extrusion, and die casting is described.

Introduction

AISI H13 is a chromium hot-work tool steel. Its chemistry is designed to withstand the temperature, pressure, abrasion, and thermal cycling associated with various hot working operations, including plastic injection molding, die casting, forging, and extrusion. The steel has low carbon content (0.4 wt%) to promote toughness, medium chromium content (5 wt%) to provide good resistance to high temperature softening, 1 wt% Si to improve high temperature oxidation resistance, and small molybdenum and vanadium additions (about 1%) that form stable carbides to increase resistance to erosive wear.

H13 tool steel plate, rod, bar, etc., is manufactured at steel mills using carefully controlled melting and manufacturing practices. The sequence of operations is equipment and energy intensive: EAF melting ⇨ ingot casting ⇨ electroslag remelting ⇨ ingot casting ⇨ soaking ⇨ preliminary forging (cogging) ⇨ reheating ⇨ forging operations ⇨ annealing ⇨

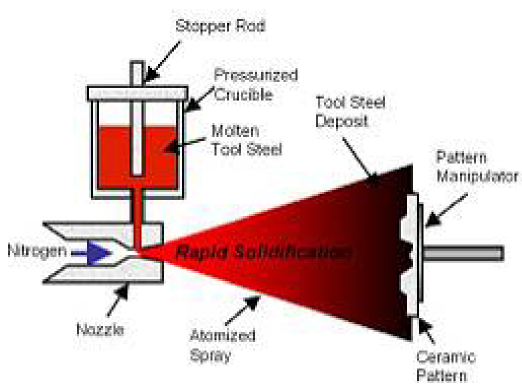
machining/cutting ⇨ bar or rod milling ⇨ annealing ⇨ finishing [Ken98]. The forged material is then converted into molds and dies using specialized machining and heat treatment operations that are also labor, equipment, and energy intensive.

Spray forming is a proven method for manufacturing molds and dies [Yan04, McH04]. Economic benefits include reduced cost, energy, and lead time due to eliminating many unit operations at the steel mill as well as most machining, benching, and heat treatment operations. Technical benefits resulting from rapid solidification include potential for die life extension. This paper examines how the cooling rate of H13 tool steel during spray forming influences phase formation, porosity, and residual stress. Die performance in manufacturing is compared with that of conventional machined dies.

Experimental

Molds and dies were spray formed using the RSP Tooling method at Idaho National Laboratory [McH04]. A summary of the processing steps is given in **Figure 1**. First, a physical model (master) was made from a computer design using a suitable rapid prototyping technology such as stereolithography. A ceramic impression was then cast in alumina. This was followed by spray forming a thick deposit of tool steel (or other alloy) on the ceramic pattern to capture the desired shape, surface texture, and detail. The deposit was cooled to room temperature and separated from the pattern. Typically, the deposit's exterior walls were machined square (or round), and bolt holes and water lines were added. The finished insert was mounted in a standard holding block or mold base.

To spray form a die, H13 tool steel was induction melted under a nitrogen atmosphere, superheated about 100°C, and pressure-fed into a bench-scale atomizer operating at 145 kPa (absolute) that was designed and constructed at Idaho National Laboratory. A nitrogen atmosphere within the spray apparatus minimized in-flight oxidation of the atomized droplets as they deposited onto alumina-based tool patterns to form deposits about 100 mm thick. Tool patterns were slurry cast under vacuum using a silicone rubber master die, or freeze cast using a stereolithography master. After setting up, the ceramic patterns were demolded, fired in a kiln, and cooled to room temperature. Deposition rates were 2.2, 2.7 and 3.0 kg/min while maintaining other parameters constant. Deposits were cooled slowly by free convection cooling in still nitrogen, or rapidly by quenching in oil or water. Higher cooling rates were achieved in some samples by forced convection cooling during deposition.



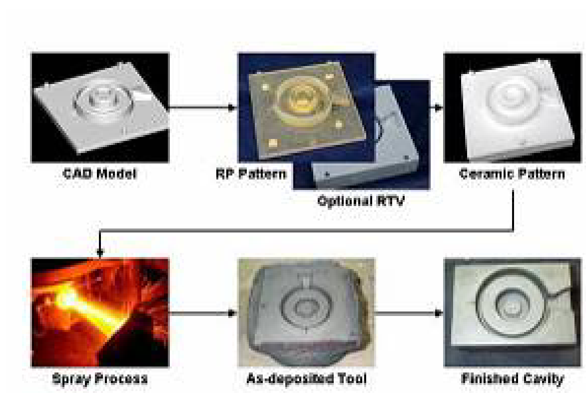


Fig. 1: Schematic of approach and processing steps

Hardness was measured using a Series 500 Wilson Rockwell C Hardness Tester by averaging eight indentations. Spray-formed material was sectioned using wire electron discharge machining and surface ground to remove the 80 µm thick heat-affected zone. Samples were heat treated in a furnace that was purged with nitrogen. Each sample was coated with boron nitride and placed in a sealed metal foil packet as a precautionary measure to prevent decarburization. Artificially aged and tempered samples were soaked 2 h or 2 h + 2 h at temperatures ranging from 500 to 630°C and air cooled. Conventionally heat-treated H13 steel was austenitized at 1010°C for 40 min, air quenched, and double tempered (2 h + 2 h) at 540°C.

Density was evaluated by water displacement using Archimedes' principle and a Mettler balance (Model AE100). Density was also evaluated using optical microscopy (OM) in conjunction with Analysis^{TM} software on polished and unetched samples. Residual stress was evaluated using neutron scattering at the National Research Council of Canada, Chalk River, ON, Canada and at Oak Ridge National Laboratory, Oak Ridge, TN, USA.

The microstructure of as-spray-formed and aged samples was analyzed by OM using an Olympus Model PME-3 metallograph, by scanning electron microscopy (SEM) using a Philips XL-30 ESEM scanning electron microscope equipped with an energy dispersive spectrometer (EDS), and by transmission electron microscopy (TEM). OM and SEM samples were etched with 3% nital solution. TEM samples were mechanically thinned to around 100 µm, and twin jet-polished using a mixture of 62 mL perchloric acid (70%), 700 mL ethanol, 100 mL butanol, and 137 mL distilled water.

Results and discussion

Chemistry

Composition analyses of spray-formed H13 and the starting material are given in **Table 1**. There is no appreciable change in composition of alloy additions after spray processing. There is, however, a small pick-up of O and a more appreciable pick-up of N. Researchers have concluded that the microstructure and properties of spray-formed H13 are not significantly influenced by small N pick-up [Sch03].

Table 1: Composition of H13 tool steel

Element	C	Mn	Cr	Mo	V	Si	N	O	Fe+other
Stock H13	0.41	0.39	5.15	1.41	0.90	1.06	0.011	0.001	Bal.
Spray-formed H13	0.41	0.38	5.10	1.42	0.9	1.08	0.059	0.002	Bal.

Porosity

Porosity in tool steels is detrimental because at high levels it can significantly reduce strength, hardness, toughness, and other properties. Sections were taken from spray-formed samples (as-deposited, aged, and conventionally heat treated) near the deposit/pattern interface, at the midsection, and near the exposed surface. Density ranged from 99.3% to 99.9% of theoretical. Density measurements from water displacement agreed with results from image analysis.

Influence of cooling rate on microstructure

The performance of H13 tool steel dies in hot-work applications depends on the microstructure, particularly at the surface of the die. Typically, an optimal microstructure consists of fine tempered martensite with few primary carbide particles. For commercial dies, this is achieved by austenitizing annealed material, rapidly cooling to below the martensite start temperature (M_s) and tempering. Austenitization allows Mo-rich M_6C and Cr-rich M_7C_3 carbides that form during annealing to redissolve. Some proeutectoid V-rich MC carbides remain because they are less soluble at austenitization temperatures [Sch87].

The microstructure of spray-formed H13 depends on spray conditions, post-deposition cooling rate, and subsequent heat treatment. Rapid cooling and solidification from near the solidus temperature (~1315°C) suppresses carbide precipitation and growth, allowing H13 dies to be artificially aged without austenitization, which can lead to unique combinations of properties. It can also lead to moderate compressive residual stresses at the surface that can reduce heat checking during service.

Figure 2 illustrates how the microstructure of H13 tool steel depends on the metal's cooling rate and processing. The structure of ingot-cast H13 (**Fig. 2a**) consists of martensite and retained austenite with extensive interdendritic segregation, coarse carbides, and shrinkage voids which result from the low cooling rate. The microstructure of commercial forged/annealed H13 that was heated to the supersolidus region (1423°C), held for 4 min at temperature, and cooled at about 1°C/min is shown in **Figure 2b**. The structure consists of martensite, retained austenite, and bainite with carbide segregates along grain boundaries. Partial melting and resolidification resulted in the formation of shrinkage voids. The presence of a liquid phase for an appreciable length of time allowed carbides to nucleate along austenite grain boundaries. During cooling, Cr in the segregate retarded the diffusion-controlled transformation of austenite to martensite [Ken98]. As with the cast structure, this structure is susceptible to brittle intergranular fracture.

Figures 2c–f illustrate the microstructure of spray-formed H13 tool steel near the center of deposits that were cooled at various rates. Samples shown in **Figures 2c, 2d** and **2f** were spray formed at 2.7 kg/min, while the sample in **Figure 2e** was produced at 3.0 kg/min. Melt superheat, gas flow rate, substrate distance, and other processing conditions were held constant. Following build-up of 50 mm-thick deposits, the samples were free-convection cooled in still nitrogen (**Fig. 2c**), oil quenched (**Fig. 2d**), or water quenched (**Fig. 2e**). The microstructures are similar despite different post-deposition cooling rates; they consisted of martensite, bainite, retained austenite, and a dispersion of spheroidal primary carbide particles. The sample in **Figure 2e** seemed to retain more austenite than the others. Coarse carbides are undesirable because they can reduce resistance to heat checking and initiate gross cracking. The sample in **Figure 2f** was cooled by forced convection with nitrogen during deposition, which resulted in a much higher solidification rate and a microstructure that was largely martensite with some grain boundary carbides and few spheroidal carbide particles. In all spray-formed samples, this structure was observed at the deposit/pattern interface due to the heat-sinking effect of the tool pattern (**Fig. 3**). This structure also formed throughout the deposit if the deposition rate was reduced to 2.2 kg/min.

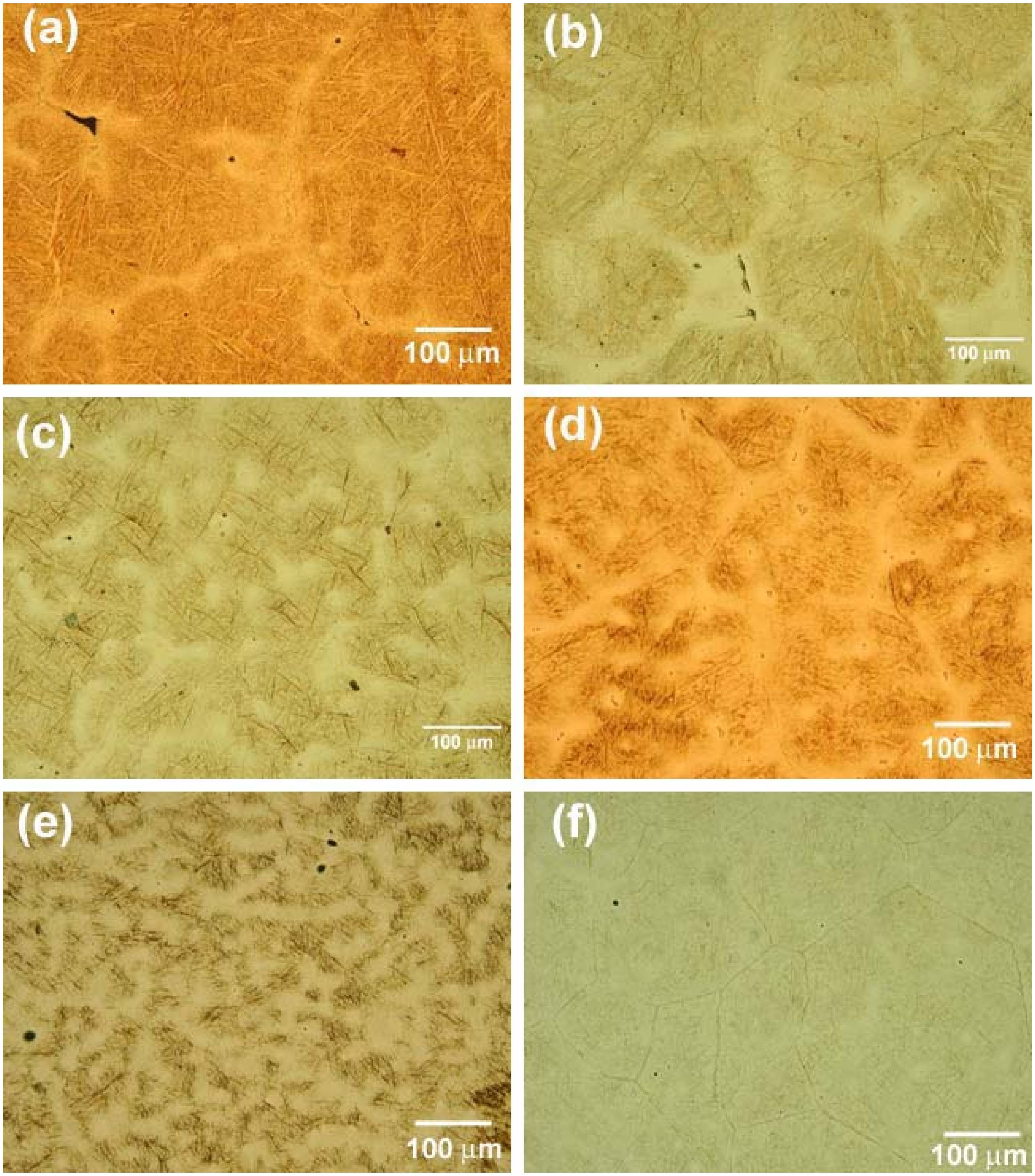


Fig. 2: Photomicrographs of H13 tool steel. All spray-formed samples were made at 2.7 kg/min. (a) Cast. (b) Commercial forged, heated to 1423°C, and cooled at rate comparable to material in (c). (c) Spray formed and free convection cooled after deposition. (d) Spray formed and oil quenched after deposition. (e) Spray formed and water quenched after deposition. (f) Spray formed and forced convection cooled during deposition.

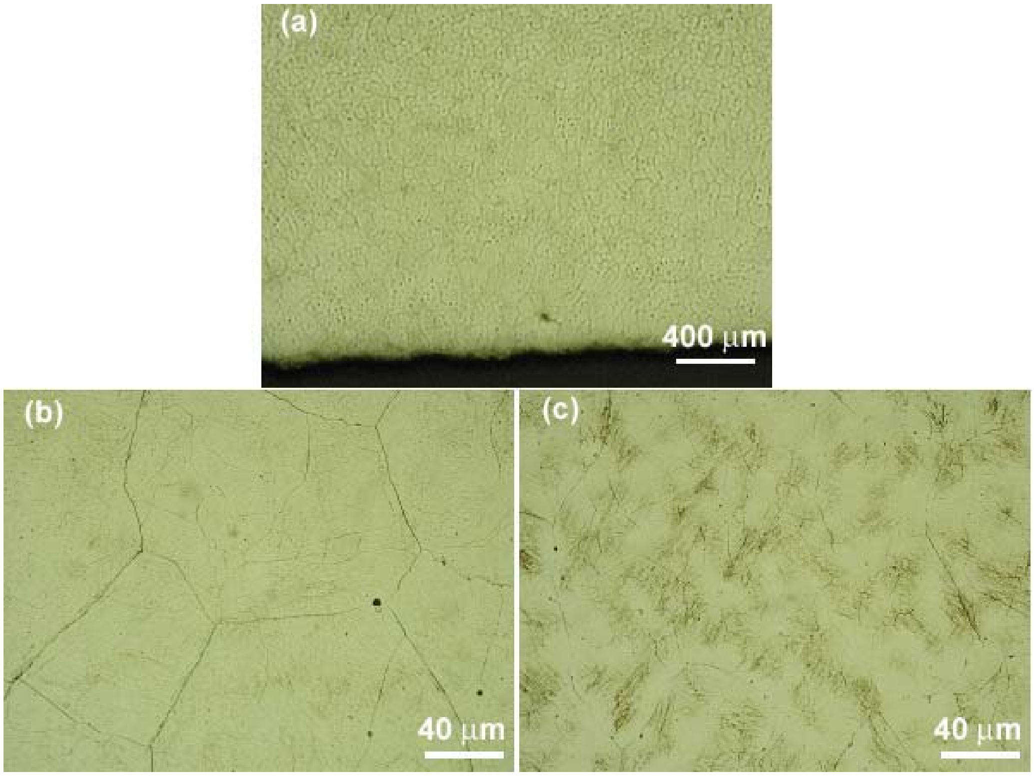


Fig. 3: Photomicrographs of spray-formed and water-quenched H13 tool steel.
(a), (b) Deposit/substrate interface. (c) Interior.

The hardness of all as-deposited H13 samples in **Figure 2** was about the same (55 HRC) and was significantly higher than that of commercial forged H13 cooled from the supersolidus region (49 HRC). The high hardness is probably due to high austenitization temperature and cooling rate. Studies with forged/annealed H13 have shown that relatively high austenitization temperatures during heat treatment increase the hardness and improve heat checking resistance despite reducing toughness [Wal84]. This is attributed to better dissolution of alloy carbides during austenitization and more uniform precipitation of fine MC carbides during tempering.

Conventional heat treatment (austenitization/quench/temper) of the spray-formed sample of **Figure 2f** generated a microstructure similar to forged H13, as shown in **Figure 4**. The microstructures consisted of a few spheroidal carbide particles in a matrix of tempered martensite. Conventional heat treatment of the samples shown in **Figures 2c, 2d,** and **2e** generated coarser tempered martensite.

The sample shown in **Figure 2f** was artificially aged. **Figure 5** illustrates the microstructure following a 2-h soak at 500°C (**Fig. 5b**), aging for 1 h at 540°C and 1 h at 580°C (**Fig 5c**), and following a 2-h soak at 650°C (**Fig. 5d**). The microstructure following a conventional heat treatment is also shown (**Fig. 5e)**. The microstructures in all these samples appear to be martensite with a few spheroidal particles of carbide.

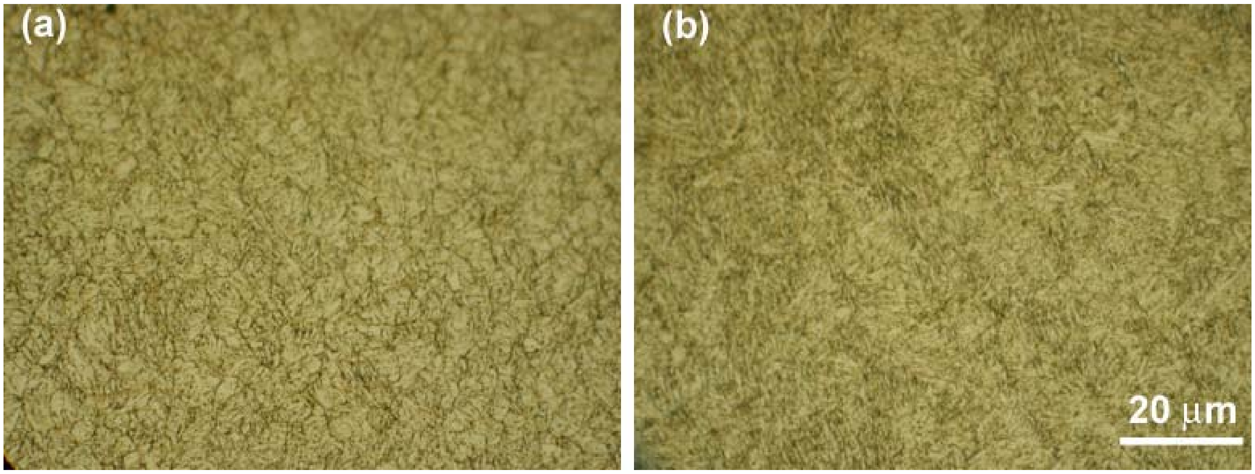


Fig. 4: Photomicrographs of H13 tool steel following a conventional heat treatment.
(a) Commercial forged. (b) Spray-formed.

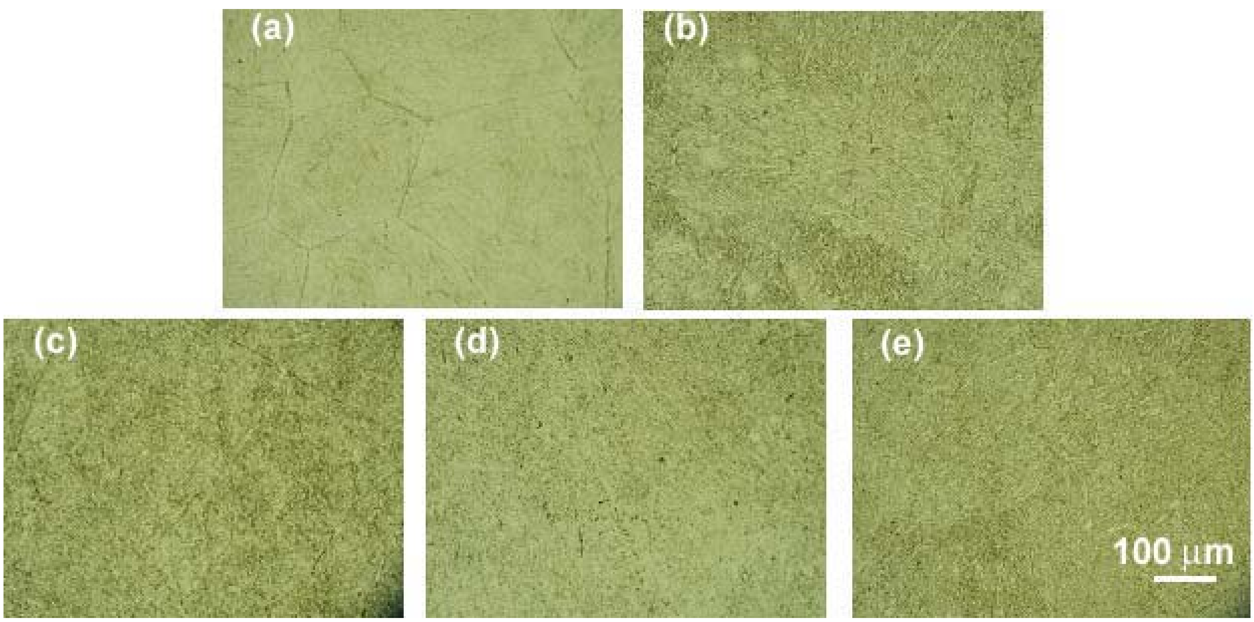


Fig. 5: Photomicrographs of spray-formed/forced convection cooled H13. (a) As-deposited.
(b) Aged at 500°C (2 h). (c) Aged at 540°C (1 h) and 580°C (1 h). (d) Aged at 650°C (2 h).
(e) Conventional heat treatment.

The optical microscope image of H13 tool steel deposited at a rate of 2.7 kg/min and cooled in still nitrogen (**Fig. 6a**) exhibits lower bainite (dark acicular features) surrounded by martensite (light regions) with patches of retained austenite (white). The SEM image, **Figure 6b**, reveals the details of these features. In Region A of **Figure 6b**, corresponding to the dark acicular features, carbides can be seen between and inside ferrite, indicating a typical lower bainite structure. Region B of **Figure 6b** corresponds to the light regions in **Figure 6a**, where martensite structure is observed. Proeutectoid carbides (V-rich MC) in retained austenite are indicated by arrows in **Figure 6b**.

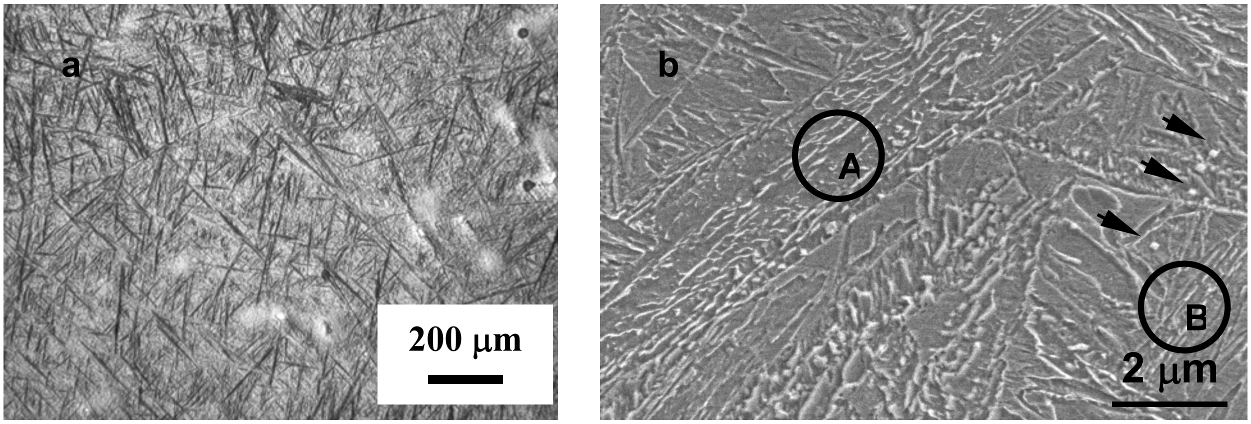


Fig. 6: Microstructures of as-deposited H13. (a) OM. (b) SEM.

The TEM bright field image in **Figure 7a** exhibits the typical lower bainite structure consisting of an aggregate of ferrite (labeled "A") and (Fe, M)C_3. Also shown in **Figure 7** are TEM bright field images of martensite (**Fig.7b**) and austenite (**Fig. 7c**). TEM analysis following aging indicates that the needle-like (Fe, M)$_3$C decomposes and the remaining (Fe, M)$_3$C exhibits either a short rod-like or a granular geometry.

Residual stress analysis

An understanding of the residual stresses that develop in a die during spray forming and heat treatment is important because stresses can affect a die's service life and dimensional accuracy. Surface stresses are particularly important. For example, large tensile residual stresses at the surface can cause gross cracking and premature heat checking during thermal cycling of a hot working die. In contrast, moderate compressive stresses at the surface are beneficial and can help prevent cracking and reduce heat checking. Large stresses can also lead to die distortion, which can result in components being formed that are out of tolerance. While a high cooling rate in tool steels can reduce segregation and improve microstructural features, it can also lead to residual stresses.

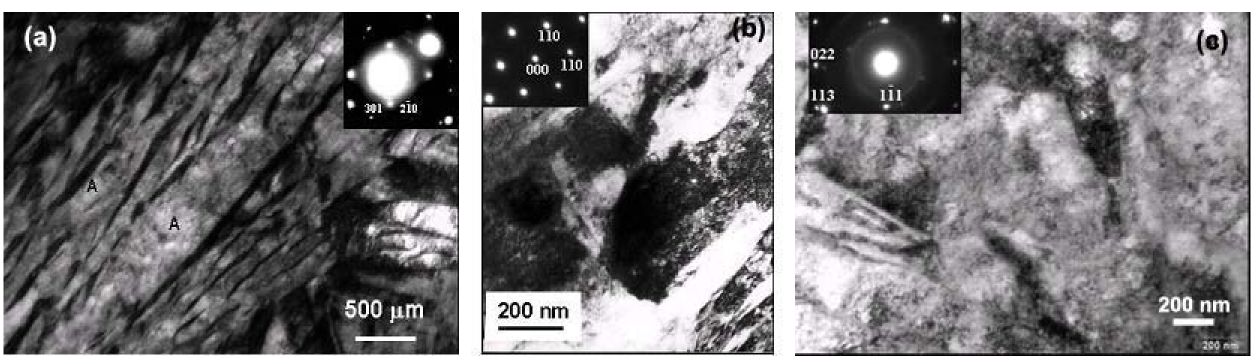


Fig. 7: TEM micrographs of spray-formed H13 of Figure 2c. (a) Bright field image of lower bainite with diffraction pattern of (Fe, M)$_3$C (inset). (b) Bright field image of martensite and diffraction pattern (inset). (c) Bright field image of retained austenite and diffraction pattern (inset).

Neutron diffraction is an ideal technique for mapping the bulk residual stress because it is nondestructive and penetrates deep into the sample. The bulk residual stresses in typical spray-formed H13 tool steel dies were measured. These measurements included three components of residual stress at different positions for several dies with different structural features. In general, surface residual stresses of the spray-formed H13 dies are compressive and transition to tensile stresses away from the surface. The magnitude usually ranges from about 100 MPa to as high as about 600 MPa [Maz98]. While fairly large, they are well below the yield strength of H13 (about 1700 MPa). High stresses can develop near some features on the tool, however. **Figure 8** shows a sample die and the measured residual stresses at one such feature. A stress relieving heat treatment is prudent, and is recommended prior to use in manufacturing. Stresses are reduced during aging or tempering heat treatments.

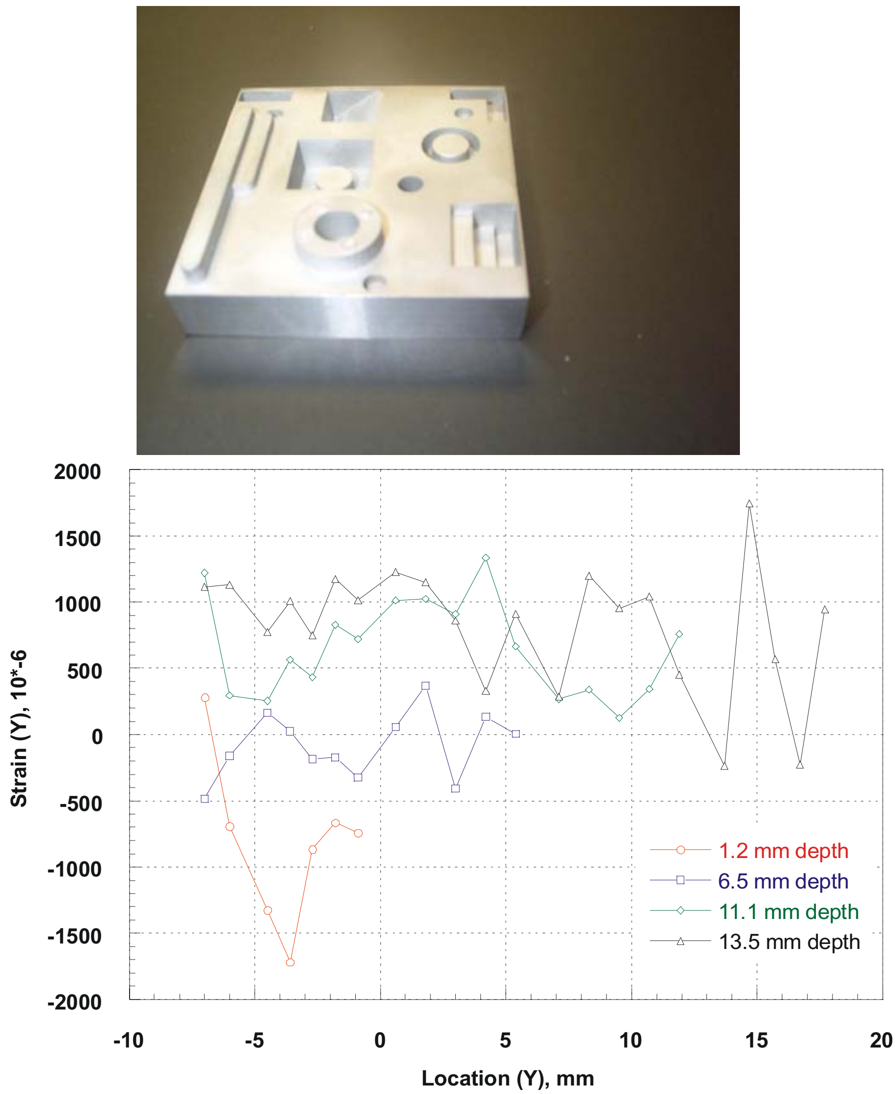


Fig. 8: H13 tool steel sample fabricated by spray forming (top); residual stresses (bottom) of the step feature in the bottom right corner of the sample.

Production runs with spray-formed H13 dies

The performance of a variety of spray-formed dies during production runs was evaluated. All runs were conducted by manufacturing companies using H13 tool steel die designs they routinely use in die casting, forging, and extrusion. Extrusion and forging dies were heat treated by aging for 2 h at 540°C. Die casting dies were also aged (540°C, 2 h + 580°C, 1.5 h). **Figure 9** summarizes the design, manufacturing process, conventional die life, spray-formed die life, and life improvement. The spray-formed dies performed well and met or exceeded the production volume of conventional dies. For example, spray-formed/aged H13 dies exhibited 20% die life extension over conventional machined dies when die casting Al alloy 380 parts. In a hot forging run of steel gears, spray-formed/aged dies were found to exhibit about 7% die life extension. When hot forging steel files, the spray-formed/aged dies had more than double the output of a conventional machined die; in another forging trial, die life was extended by 67%. Finally, in a cold-extrusion study of H13 bar, as-spray-formed dies outlasted conventional H13 dies by 50%. Aluminum die casting, forging, and extrusion are three of the most demanding applications of commercial dies. Performance studies in other forming operations are ongoing.

Die Design	Process	Machined Die Life	Spray Formed Die Life	% Improvement
	Forging	2500	2675	7%
	Extrusion	20	30	50%
	Forging	2500	7000	180%
	Forging	3000	5000	67%
	Die Casting	85000	102000	20%

Fig. 9: Summary of die life studies in forging, extrusion, and die casting H13 tool steel dies.

Conclusions

1. Quenching spray-formed H13 tool steel after solidification (oil, water, or free convection) was less effective at refining microstructural features at the midsection of dies than was quenching the material during deposition by forced convection cooling. The former resulted in a combination of martensite, lower bainite, retained austenite, and spheroidal primary carbides while the latter resulted primarily in martensite with few primary carbides. This structure is also obtained at the deposit/pattern interface due to the higher solidification rate at the interface.
2. Density of spray-formed H13 dies ranged from 99.3% to 99.9% of theoretical.
3. Residual stresses that form during deposition depend on processing conditions and the geometry of the tool pattern. The stress state of a die is typically compressive at the deposit/substrate interface and tensile at the midsection.
4. Spray-formed and aged H13 tool steel dies exhibited longer die life than conventional forged and heat-treated dies in production runs in aluminum die casting, forging, and extrusion.

Acknowledgements

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