



PII S1359-6462(98)00349-2

## RESIDUAL STRESSES AND MICROSTRUCTURE OF H13 STEEL FORMED BY COMBINING TWO DIFFERENT DIRECT FABRICATION METHODS

P.J. Maziasz<sup>1</sup>, E.A. Payzant<sup>1</sup>, M.E. Schlienger<sup>2</sup> and K.M. McHugh<sup>3</sup><sup>1</sup>Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6115 <sup>2</sup>Sandia National Laboratory, P.O. Box 5800, Albuquerque, NM 87185-1134 <sup>3</sup>Idaho National Engineering and Environmental Laboratory, P.O. Box 1625, Idaho Falls, ID 83415-2050

(Received August 5, 1998)

(Accepted August 11, 1998)

### Introduction

Direct fabrication (DF) of tool and die steels by rapid solidification techniques can produce near-net-shape parts and components with unique properties, and without the distortions caused by conventional normalizing and tempering heat-treatments [1,2]. When combined with sophisticated 3-dimensional computer control to build complex solid metallic shapes, one has the capability of using DF for rapid prototyping [3]. Spray forming using a circular converging/diverging atomizer is a DF process being developed at the Idaho National Engineering and Environmental Laboratory (INEEL) for rapid manufacturing of tool and die steels like H-13 [2]. Laser Engineered Net Shaping (LENS<sup>TM</sup>) is a DF process being developed at Sandia National Laboratory (SNL). LENS involves laser-processing fine powder metal sprays into complex, fully-dense 3-dimensional shapes with fine-detail control that would allow rapid prototyping of tools or dies. One logical combination of the two processes is to combine spray forming to replicate most of the die surface and backing, and then to build other die-surface fine-features with LENS. Premium H-13 steel was used because it belongs to the widely used group of hot-work steels that have good resistance to heat, pressure and abrasion for metal-forging and aluminum die-casting applications [4]. The microstructure and residual stresses that exist across the interface of a composite metal produced by these two DF methods are critical parameters in producing crack-free components with functional properties. Most techniques for measuring residual stresses are based on X-ray diffraction, and are, therefore, confined to near-surface measurements [5,6]. The purpose of this work is to combine unique neutron-diffraction facilities at the Oak Ridge National Laboratory (ORNL) for measuring bulk residual stresses [6] with these two different DF processes to characterize LENS deposits of H-13 steel made on a spray-formed base of that same steel.

### Experimental

Details of the LENS and the INEEL spray forming DF processes have been described elsewhere [2,3], and will be summarized here. The composite DF specimen of H-13 steel, shown in Fig. 1, was obtained by producing the initial spray-formed material, and depositing H-13 steel with LENS after two different surface preparations. LENS H-13 steel was deposited on the as-received spray-formed surface, and on

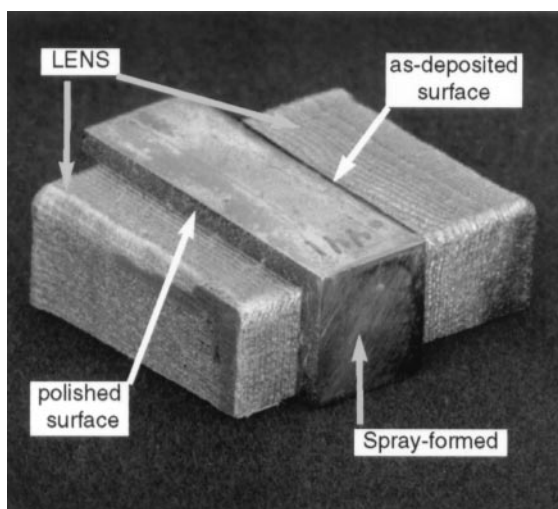


Figure 1. LENS deposits of H-13 die-steel made on a spray-formed piece of the same steel, but with two different surface conditions.

a mechanically-polished surface, as shown in Fig. 1. The spray formed H-13 steel block was produced by induction melting H-13 in a nitrogen atmosphere (100°C superheat), and pressure-feeding it into a bench-scale circular converging/diverging spray nozzle. The spray deposit builds up on an ambient steel plate in an inert gas atmosphere at a rate of 200 kg/h. The LENS deposits were made using streams of H-13 steel powder injected into a focussed laser beam, in a glove box with an argon atmosphere. The LENS platform is a 1.8 kW cw Nd:YAG laser in a glove box with powder feed system a computer controlled positioning target stage; these LENS H-13 steel deposits were made at a laser power of 280 W. Both DF processes used H-13 steel produced by Crucible, and chemical analyses were done on similar, monolithic deposits from each technique. The LENS H-13 was 0.38C-0.36Mn-5.34Cr-1.48Mo-1.02V-1.04Si-bal.Fe (wt.%), and the spray formed H-13 was 0.41C-0.38Mn-5.1Cr-1.42Mo-0.91V-1.08Si-bal.Fe. Both are within specifications for premium-grade commercial H-13 steel.

Monolithic individual DF deposits and the composite DF deposit were characterized at ORNL, using metallographic, hardness (Vickers, Hv, and conversion to Rockwell C, HRC), and neutron-diffraction residual stress measurement techniques. Metallographic specimens were diamond-polished and etched with 2% picric acid in ethanol solution. Hardness was measured on monolithic deposits with a computer-controlled Beuhler Micromet 2001 Microhardness Tester. Neutron diffraction was used to measure residual strains, from which residual stresses were calculated. Strain profiling was done using the modified, triple-axis neutron spectrometer at the High Flux Isotope Reactor (HFIR) at ORNL [6]. Apertures defined the beam-analyzed volume, and strains normal, transverse and parallel to the composite DF interfaces were measured along the specimen axis. Strains were also measured in monolithic, bulk DF deposit specimens, and stress-relieved  $\frac{1}{3}$  Charpy specimens of each were used as stress-free references.

### **Results and Discussion**

Metallographic analysis shows very different native microstructures in monolithic LENS and spray-formed deposits of H-13 steel (Fig. 2). The spray formed H-13 has a characteristic equiaxed, fine-grained bainitic structure with some retained austenite at grain boundaries and general porosity.

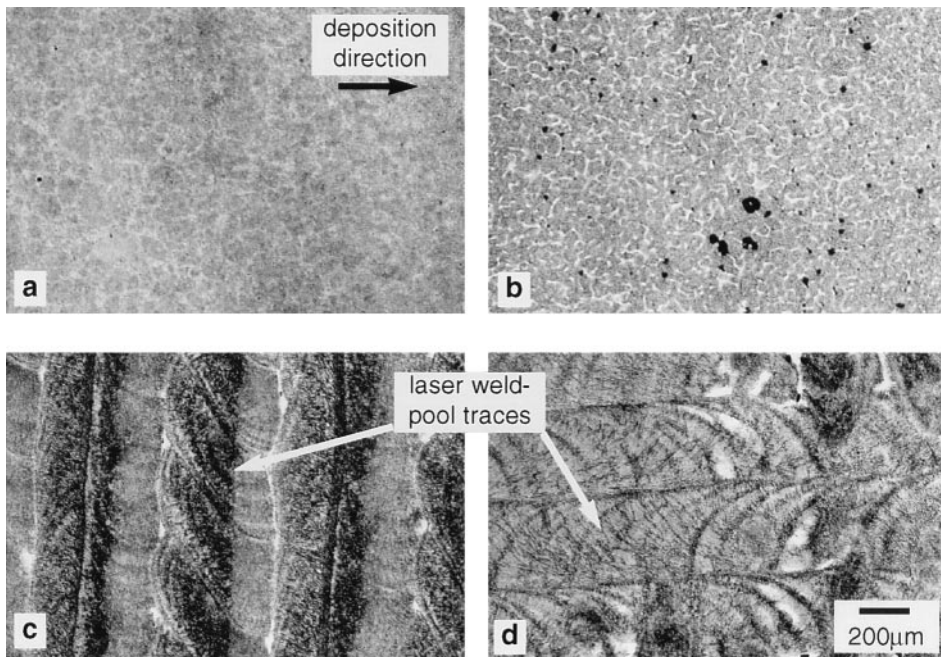


Figure 2. Characteristic microstructures of spray-formed H-13 steel a) at the as-sprayed surface, and b) deeper into the spray-deposit, and of LENS H-13 steel c) showing layers along the side, all viewed in sections cut parallel to the deposition direction. d) shows the laser patterns on a section cut perpendicular to the deposition build-up direction.

Despite traces of micro-porosity, the spray-formed H-13 steel was measured to have 98% of theoretical density. Porosity and retained austenite are least near the surface (last material deposited) (Fig. 2a and 2b). The LENS H-13 has a finer, more uniform martensitic structure, with possibly some retained austenite. The characteristic rows and layers of the LENS material reflect solidification from the moving laser-weld pool (Fig. 2c and 2d). LENS H-13 steel has an average as-deposited hardness of 564 Hv (52 HRC), whereas the spray-formed H-13 has a hardness of 659 Hv (61 HRC). Typically, premium tempered H-13 is used with hardnesses of 410–485 Hv (42–48 HRC). The nature of the two H-13 steels produced by each DF method is quite different, however, because when they are both tempered for 2h at 570°C, the spray-formed steel softens to 574 Hv (53 HRC) whereas the LENS steel hardens to 662 Hv (61 HRC).

The composite specimen of LENS deposited on spray-formed H-13 steel shows an interfacial structure and “processing zone” that differ from the characteristic “signature” microstructure of each DF process (Fig. 3). This “processing zone” interface is also significantly affected by the surface condition of the spray-formed material. For LENS deposited on as-received spray-formed material, the total processing zone is about 500  $\mu\text{m}$ , with a 200  $\mu\text{m}$  layer that contains some lower bainite and retained austenite and a 300  $\mu\text{m}$  overtempered layer between the LENS and spray-formed steels (Fig. 3a and 3c). For LENS deposited on mechanically-polished spray-formed steel, the “processing zone” at the interface is wider (650  $\mu\text{m}$ ), with a 300  $\mu\text{m}$  region with clear, coarse prior-austenite grains visible and a 350  $\mu\text{m}$  overtempered layer between the LENS and spray-formed steels (Fig. 3b and 3d).

Neutron diffraction measurements of residual strains and stresses mapped along the axis of the composite DF specimen are shown in Fig. 4. The stresses are referenced relative to the interfaces between the spray-formed and LENS deposits, as indicated in the coordinate system diagram in Fig. 4. The composite specimen has large compressive normal stresses in the spray-formed material, which

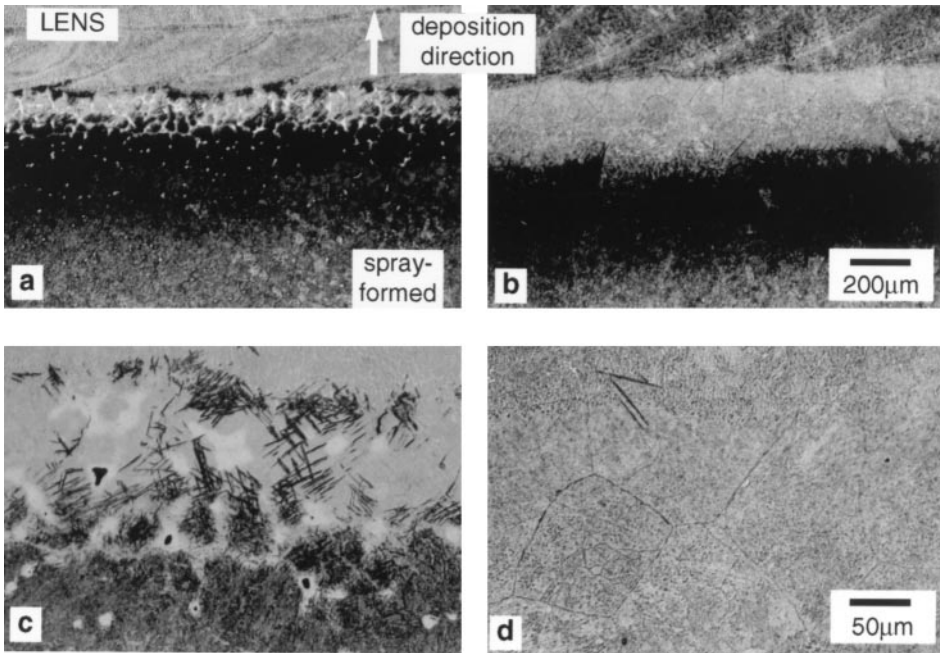


Figure 3. Microstructure of the interfacial structure developed between spray-formed and LENS H-13 steel deposits, with a) and b) at lower magnification and c) and d) at higher magnification. a) and c) are for LENS deposited on the final as-sprayed surface and b) and d) are LENS deposited on a mechanically-polished surface deeper into the spray-formed material. These interfacial regions are different than either monolithic steel.

decrease or reverse in either of the LENS deposits. In the LENS material deposited on the as-received spray-formed steel, the normal stress is lower at the interface and then approaches zero farther into the LENS material. By contrast, the normal stress reverses across the mechanically-polished interface and becomes highly tensile, before declining toward zero in the LENS deposit. The normal stress difference across the mechanically-polished interface is close to 900 MPa. Despite such a large stress, H-13 steel typically has a yield stress of about 1700 MPa, so even these stresses are still within the elastic region of the material. The longitudinal stress is different, and is near zero at the mechanically polished interface, and is slightly tensile at the other interface. The transverse stress somewhat reflects the normal

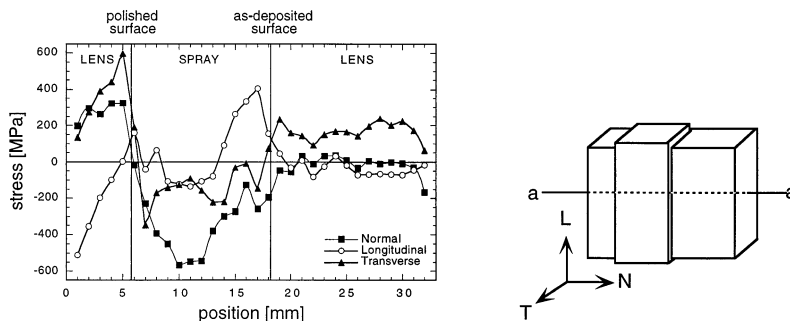


Figure 4. Three orthogonal components (T, L, N) of residual stress were determined along line a-a in the LENS-SPRAY-LENS DF H-13 steel composite specimen. Results are value averaged so that actual peak stress values may be higher.

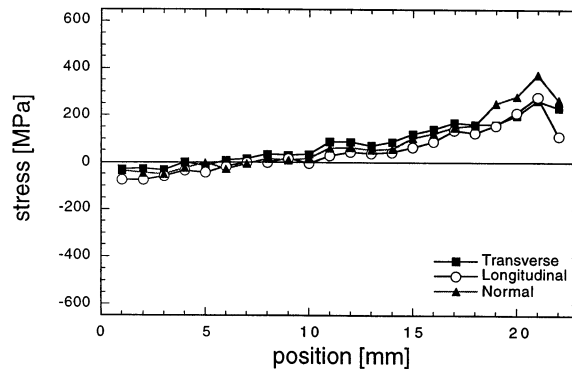


Figure 5. Three orthogonal components (T, L, N) of residual stress along an equivalent line a-a in a monolithic LENS H-13 steel deposit, with deposition build-up in the normal direction, and position = 23mm marking the surface with final deposition. The apparent tensile stress near the final surface may also reflect changes in the lattice parameter caused by microstructural differences due to the final heat-treatment in that region.

stress, and is slightly compressive in the spray formed steel before becoming tensile in both LENS deposits. The stress reversal near the as-deposited interface is about 400 MPa, whereas the stress change across the mechanically-polished interface is close to 1000 MPa. No cracking was observed at either interface. Clearly stresses are worse at the mechanically-polished interface which has the thicker “processing zone” than at the unpolished interface with the narrower “processing zone.” If the material at the mechanically-polished interface was heated into the austenite region, as the microstructure suggests, then those additional transformation stresses could also contribute to the higher residual stresses found at that interface.

Residual stresses in the monolithic LENS H-13 steel specimen are somewhat less than those found at the interfaces of the composite DF specimen (Fig. 5). There were almost no residual stresses in the interior of the LENS H-13 steel specimen, and then tensile stress increased to nearly 400 MPa beneath the surface deposited last. This is consistent with the heat from new LENS layers stress-relieving the material beneath. By contrast, very large tensile stresses appear to be present in the middle of the large, monolithic spray-formed H-13 deposit, which then diminish to about 400 MPa near the surface of the last material deposited. In this monolithic spray-formed specimen, repeat direct neutron diffraction measurements of the lattice parameter gave the same lattice parameter data, but interpreting those lattice parameter differences as residual strains and stresses depends on the assumptions that the microstructure and microcomposition of the steel stay the same. In the case of the spray-formed material, there are clearly depth-dependent changes in microstructure (Fig. 2a and 2b) compared to the more uniform structure of the LENS material. A difference in carbide precipitation or the relative fraction of retained austenite in the spray-formed material could affect such residual stress measurements, so more characterization and neutron diffraction data are needed to better understand the spray-formed H-13 material. However, it is clear from these results that large residual stresses can exist at the interfaces of materials formed by combining two different DF processes, and that high-strength materials like H-13 tool steels can retain large elastic stresses after processing.

### Summary

Direct fabrication techniques can be combined to produce composite DF specimens, with spray-forming rapidly building up the base material for die-surface features and backing, and LENS creating the other

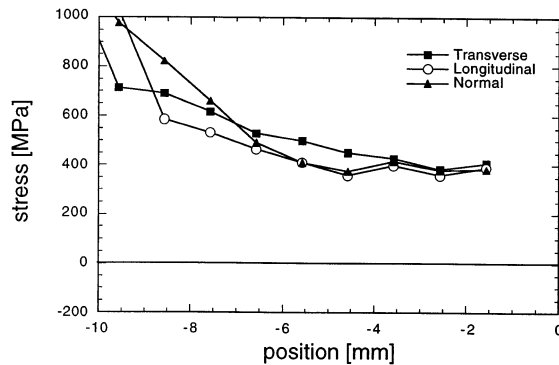


Figure 6. Three orthogonal components (T, L, N) of residual stress along an equivalent line a-a in the monolithic spray-formed H-13 specimen, with position = 0mm marking the surface of final deposition. The data was confirmed by repeated measurements. Depth terminates in the middle of the original spray-formed deposit, and corresponds to about the same position as the polished interface in Fig. 4. The similar relative behavior of the different stress components suggests that the apparent hydrostatic stress is ore likely a lattice parameter difference between the reference sample and the bulk sample caused by compositional variations during rapid forming.

final-surface or fine-detail features. Residual stresses are lower, and the microstructural “processing zone” that marks the transition between the characteristic structure of each native DF process is narrower, for LENS deposited directly on the final as-spray-formed surface instead of a mechanically polished surface. Stress-relief of the spray-formed H-13 steel prior to additional LENS processing may also lower the residual stresses across the transition interface. Very large residual stresses can exist in H-13 steel across the interface between the two inherently different DF processes. However, proper characterization feedback should allow surface preparation and heat-treatment parameters to be chosen which minimize such stresses.

### Acknowledgments

Thanks to E.H. Lee for metallography of the various H-13 steel specimens. Research sponsored by the Laboratory Directed Research and Development Program of the Oak Ridge National Laboratory, and in part by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies as part of the High Temperature Materials Laboratory User Program at the Oak Ridge National Laboratory and also by the Division of Materials Science, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp.

### References

1. N. J. Grant, *Metall. Trans. A*, 23A, 1083–1093 (1992).
2. K. M. McHugh, in *Solidification 1998*, ed. S. P. Marsh et al., pp. 427–438, TMS, Warrendale, PA (1998).
3. D. M. Keicher et al., in *Advances in Powder Metallurgy and Particulate Materials—1996*, vol. 4, part 15, pp. 15–119–15–127, MPIF, Princeton, NJ (1996).
4. Hot-Work Tool Steels, in *Tool Steels, 5th edn.*, ed. G. Roberts, G. Krauss, and R. Kennedy, pp. 219–250, ASM International, Materials Park, OH (1998).
5. S. Iwanaga, *Transactions of the 19th Die Casting Congress and Exposition*, pp. 213–219, NADCA, Rosemont, IL (1997).
6. X.-L. Wang et al., *Mater. Sci. Eng. A211*, 45–53 (1996).