

Change in hardness and microstructure during cumulative heating of tool steel H13

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Hybrid-structured dies are possible to fabricate with developing Laser-Melt-Deposition (LMD) technique. A 3D fabrication of tool steel (H13), however, usually results in an inhomogeneous hardness due to the heat affections during multi-layer deposition. In order to assess such cumulative heating, we have studied two kinds of tool steel: a standard H13 and a modified steel with low Cr, Si content (LCS).

Starting materials were prepared by remelting the two steel pieces as for "as-deposited" state. The solidified samples were then induction-heated cumulatively at the rate of 10K/s to fixed temperatures from 1473 to 573K. After each heating step, hardness was measured and critical change in microstructure was analyzed in detail by SEM and EDS analysis on electrically extracted residue.

Descending the heating temperature, the two steels exhibited a discontinuous softening after heating to 973-1093K, and still the LCS kept a high hardness of 450Hv even after heating to temperatures under 973K. Fine precipitates with several tens of nanometer were observed in the LCS heated to 973K. By cooling from austenite region (1273K), the microstructures was martensite and MC-type carbide. In the subsequent heating to ferrite-austenite region, both reverse transformation to austenite and carbide precipitation can take place.

Thermodynamic calculations suggested the resultant hardness relies on a competition of M_7C_3 precipitation and reverse transformation. In low-Cr or low-Si tool steels, partial γ transformation took place on heating, which suppressed softening by fresh martensite and nano-scale precipitates.

Keywords: laser metal deposition, hardness, reverse transformation, martensite, carbide precipitation

1. Introduction

The tool steel H13 (SKD61 in JIS) has been widely used for high-temperature forming die and the dies are repaired by plasma overlaying with the H13 corresponding wires. Recent advances in laser 3D fabrication technique lead challenges to produce hollow die parts¹⁾ and hybrid structures²⁾ for accelerated die cooling. Laser metal deposition (LMD), a developed form of laser overlaying, has a higher building rate and suitable features for additive manufacturing compared with Selective Laser Melting (SLM). On the other hand, LMD process has intrinsic problems such as the effect of large heat input and insufficient dimension accuracy.

As a laser process for hybridizing the authors has proposed the bonding-building of H13 onto a thick Cu plate, and succeeded in the fabrication of 7-layer-deposit bead with dimensions of about 8mm wide and height³⁾. An improvement of cooling efficiency was reported elsewhere⁴⁾, and the hardness of interest showed an inhomogeneous distribution of 550~670Hv from lower to upper deposit layers.

In response this, one of the author (Y. K.) carried out post heat treatment on the H13 deposit and low-alloyed tool steel deposit⁵⁾. The degree of softening was larger in the latter resulting in more homogeneous hardness around 500Hv.

The "as-deposit" hardness is mostly high due to the significant hardenability of the steel. It is then affected by heat input from every deposition of upper layers. In lower layers of a deposit the heat affections are cumulative and the peak temperature may change from austenite (γ) region to ferrite (α)+carbide tempering range. For this complexity it is difficult to control the hardness of LMD deposit based on a known tempering-softening curve.

In this work the relation between hardness change and

microstructure during cumulative heating has been investigated. The softening behaviors with descending temperatures were compared for two different tool steels and discussed on the basis of thermodynamic considerations.

2. Experimental Procedure

2.1 Preparation of material

A JIS-based SKD61: SKD and the modified tool steel with low Cr and low Si content: LCS were used in experiments. The typical compositions are given in Table 1.

Table 1 Composition of test steels (mass%)

	C	Si	Mn	Cr	Mo	V	Fe
SKD	0.4	1.0	0.4	4.9	1.2	1.0	Bal.
LCS	0.3	0.1	1.6	1.9	1.0	0.6	Bal.

For the starting materials, 10g of sample steels were induction-melt at temperatures above 1873K and poured into a refractory die. The as-cast specimen was about 8mm in diameter and hereafter called "as-depo" to mean as-deposit state before receiving any heat affections.

2.2 Cumulative heat treatment

Referring to the phase diagram for H13, the *as-depo* samples were cumulatively heat treated to peak temperature in γ (1473K), γ +VC (1273K), γ + α (1093K), α (973K) ranges, and thereafter temperatures in descending order with 100K interval. Temperatures were controlled with a spot-weld thermocouple (type K) on the sample surface so as to be heated at 10K/s and held for 10s in Ar-filled chamber. Every time after natural cooling, the samples were subjected to hardness measurement with Vicker's microhardness tester (500gf).

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2.3 Microstructure analysis

Microstructures were investigated on samples showing a significant change in hardness before/after a specific heating. Polished surfaces were chemically etched and observed in scanning electron microscopy (SEM). Precipitates were extracted as residues from electrolytic method using 10% AA (acetylaceton ammonium chloride) methanol solution. The ratio of metallic elements in precipitates was averaged for ten analysis points in energy-dispersive spectroscopy (EDS).

3. Experimental Results

3.1 Change in hardness

Figure 1 compares hardness change of the two *as-depo* steels measured after each step of the cumulative heating. In both steels, the samples heated to 1473K show 600Hv equivalent to as-quenched hardness, which decrease gradually on subsequent heatings.

The hardness of H13 decreases significantly after 1093K heating, while that of LCS shows a discontinuous decrease after 973K heating. It is interesting that the degree of softening is larger in H13 than in LCS having lower Cr and Si content. The hardness of LCS remains around 450Hv with no further decrease until 573K heating. This difference would be interpreted in view of tempering of martensitic microstructure.

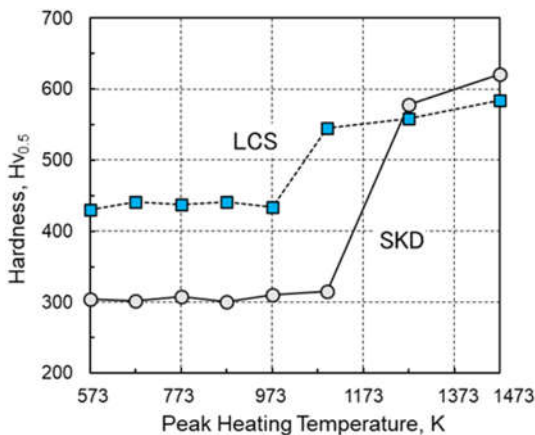


Figure 1 Change in hardness of *as-depo* samples after the cumulative heating from 1473K to 573K.

3.2 Change in microstructures

Figure 2 (a) represents a martensitic microstructure observed in both steel samples heated to 1273K. Very fine particles are observed within martensite plates and it was identified VC from (b) the ratio of metallic elements of extracted residue. The slight decrease in hardness after 1273K heating is attributed to a decrease in C concentration in γ due to VC precipitation.

As shown in Figure 3 the martensite morphologies for the two steels changes in different ways after 1093K heating. Non-acicular features in (a) H13 are considered to those of tempered martensite, and numerous facet particles of 50~150nm are precipitated on some boundary surface.

On the other hand in (b) LCS, heated to the same temperature, acicular regions of about 20 microns are randomly distributed, which occupied nearly a half of

microstructures. One of the reason for the higher hardness (540Hv) was thought to be these martensitic islands which was newly transformed on cooling from 1093K. Flakes found on its linear boundaries should be some carbide which has been precipitated with the martensite formation.

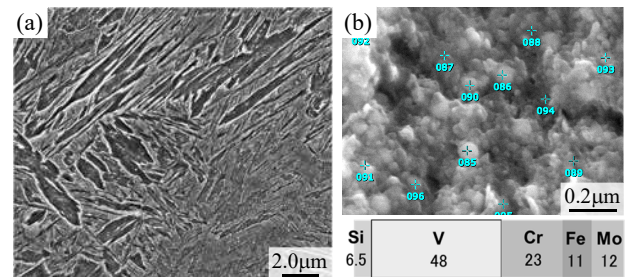


Figure 2 (a) Microstructure of H13 cumulatively heated to 1473K → 1273K. (b) Metallic elements (at%) in extracted residue.

3.3 Carbide precipitation

In high magnifications in Figure 3 the difference in precipitates are clear and, in LCS, dense nano-scale particles are also observed other than the flakey carbide. The nano-scale particles were more evidently observed after 973K heating, which therefore was responsible for the higher hardness thereafter.

The metallic elements in extracted precipitates are compared in Figure 4. Equilibrium ratio of metallic elements

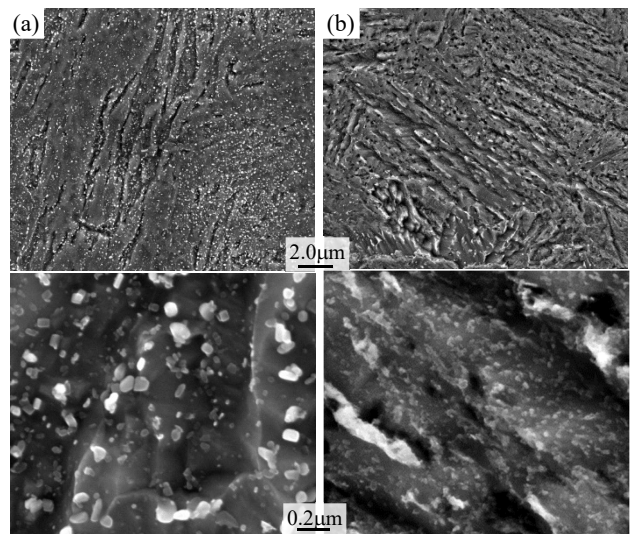


Figure 3 Microstructures heated to 1473→1273→1093K and (below) high magnification images of precipitates (a) SKD (b) LCS.

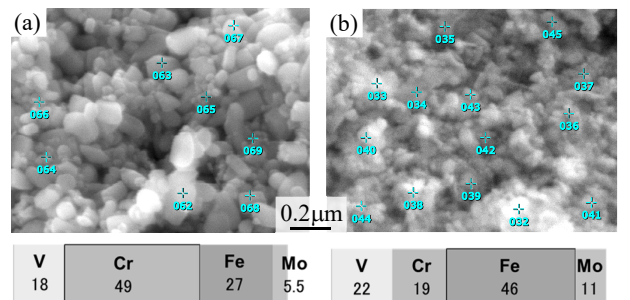


Figure 4 Images of extracted precipitates and ratio of metallic elements by EDS analysis and equilibrium calculation. (a) H13 after 1093K heating and (b) LCS after 973K heating.

are also calculated assuming the precipitates as M_7C_3 for H13, $M_{23}C_6$ for LCS. After 1093K heating, the hardness of H13 decreased to 310Hv and (a) the precipitates contains 49at% of Cr, and Fe, V. On the other hand, the hardness remains high in LCS and the fine precipitates were V-rich similar to those in Figure 2(b).

This precipitates constituent changes in (c) 973K-heated sample in which the nano-scale particles are clearly seen. It consists of 46 at% Fe and 19% Cr, which is too small Cr ratio for M_7C_3 . So this Fe-rich precipitates are the mixture of MC and $M_{23}C_6$ or M_3C as candidate carbide.

4. Discussion

In this section, the difference in microstructure of two steels has been discussed focusing on the samples heated to $\gamma+\alpha$ region. As shown in Figure 4, precipitated carbides were different in H13/LCS after the significant softening occur respectively in 1093K/973K heating.

For the alloy composition of H13, M_7C_3 is in equilibrium at around 1123K and replaced by $M_{23}C_6$ below 1093K. In a rapid heating of the sample consisting martensite+VC (Figure 2), precipitation of M_7C_3 would be competitive with α' to γ reverse transformation. Moreover M_7C_3 is less stable in the low-Cr steel; LCS.

Assuming M_7C_3 would not precipitate in a rapid heating, the metastable phase diagrams were calculated in Figure 5, where open circles mark the C content of the two steels. At 1093K H13 is located in $\alpha+MC+M_{23}C_6$, while LCS in $\gamma+\alpha+MC$. It suggests that, for low-Cr steel, α' to γ transformation can take place on heating in preference of M_7C_3 precipitation. Though these two cases are both extreme cases, the possible γ transformation is caused by reduced stability of M_7C_3 and descending temperature of $\gamma+\alpha$ region.

For further discussion, the equilibrium composition of austenite at 1273K was assumed to go to martensite and then equilibrium phases were evaluated for 1103K heating (for convenience, 10K higher than the experimental). In this calculation, a hypothetical alloy 5Cr-0.1Si was also evaluated to see the single effect of Si content.

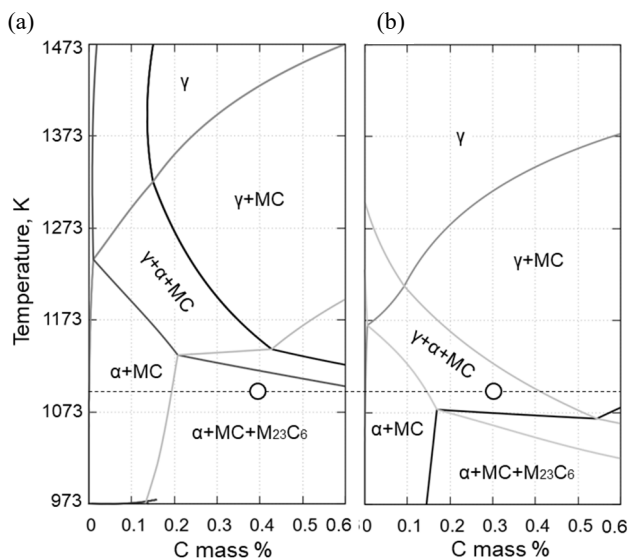


Figure 5 Metastable phase diagram for (a) H13 and (b) LCS excluding M_7C_3 with calculation condition of suppressed phase.

The result is graphically shown in Figure 6. Not only LCS (2Cr-0.1Si), but also in the case of low Si (1/10 of H13), more than a half of equilibrium phase is γ . And if this partial γ transforms to fresh α' , large driving force for carbide precipitation is expected because of higher C concentration than the alloy average. For the case of LCS, the calculated driving force for M_3C precipitation is the highest in the 973K next heating. Though the nano-scale particle has not been clearly identified, this large driving force explains the fineness of the particles and the less softening behavior of LCS.

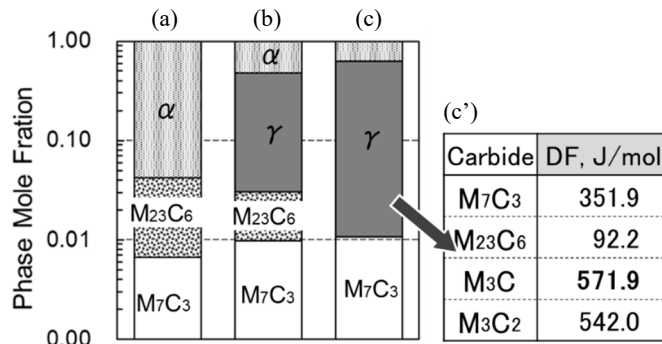


Figure 6 Equilibrium phase fraction of (a) SKD 5Cr-1.0Si, (b) 5Cr-0.1Si, (c) LCS 2Cr-0.1Si at 1103K based on the equilibrium composition of austenite at 1273K. (c') Driving force for precipitation at 973K in α' transformed from partial γ in (c).

5. Summary

- (1) In the cumulative heatings, *as-depo* samples showed a discontinuous decrease after heated to 1093K for SKD and 973K for LCS. The degree of softening was larger in SKD than in LCS.
- (2) The LCS kept a high hardness of 450Hv even after heating to temperatures under 973K, in which nano-scale particles were observed.
- (3) Thermodynamic consideration revealed that, in low-Cr or low-Si tool steels, partial γ transformation took place on heating, which suppressed softening by formation of fresh martensite and precipitation of nano-scale particles.

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References

- 1) M. Kato, J. Umeda, Y. Fukuta, T. Ikeguchi, J. Kato: Research Report of Aichi Center for Industry and Science Technology (2019) pp.14-17.
- 2) U. Articek, M. Milfelner, I. Anzel: Adv. Prod. Eng. Manage. **8** (2013) No.3 169-176.
- 3) K. Tanaka, Y. Oka, T. Sugimoto: J. Japan Inst. for Copper **60** (2021) No.1 50-55.
- 4) K. Tanaka: Research Report of R&D grant of Sango Monozukuri Foundation (2022), *online* https://sango-mono.or.jp/?page_id=344.
- 5) Y. Kodama and K. Tanaka: Abstracts of the 12th Meeting of the Japan Soc. Heat Treatment, Chubu Branch (2022) pp.3-4.