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The TEM study of structure of 1H18N9T stainless steel buildups

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ABSTRACT

Purpose: The reason of investigation was finding the answer of the question what was the reason of extreme hardness of build-ups created during bulk metal forming processes of austenitic stainless steel.
Design/methodology/approach: The studies were conducted using transmission electron microscopy (TEM). Since very specific specimens special method for thin foil preparation were elaborated. Thin foil method was chose because it was only one which supply both structure and diffraction information.
Findings: On the basis of careful analysis structural and electron diffraction pattern it was proposed that simultaneously to very well known strengthening mechanisms like grain refinement and dislocation density increase, the additional mechanism can not be excluded. Based mostly on the electron diffraction pattern (appearance of forbidden reflexes for FCC) it is proposed that this additional mechanism could be the marthensitic transformation caused by very high plastic deformation.
Practical implications: At this moment it is difficult to point out the practical implications of the result however the mechanism proposed in this elaboration might be used in design of high strength materials.
Originality/value: The main achievement of this investigation is TEM results which analysis allow to propose that an extra-mechanism for strengthening of 1H18N9 stainless steel is marthensitic transformation involved with high plastic deformation in build-ups. Additionally the special method of thin foils preparation was elaborated.
Keywords: Plastic forming; Build-ups; Microstructure

1. Introduction

In metal forming processes in the tool-workpiece contact area occur high pressure, surface expansion and elevated temperature. It makes ideal circumstances for braking of the lubricant film that causes an direct contact between metallic surfaces. Such a contact usually leads to building connecting bridges and successively growing of buildups on the tool surface [1,2]. These phenomena that mostly result very big surface damages are called galling. Although the consequences of galling are easy to observe, process itself and its parameters are still not very well known. In the case of metal-forming products, because of their functional requirements, the effects of galling are unacceptable. To stop or at least limit galling tendency many different methods are investigated like tool surface modifications [3,4], workpiece roughness control [5] or tool vibrations [6]. Galling also causes process increasing of forces and can even lead to tool surface damages. It is possible because buildups although created from rather soft billet material are itself very hard. Their hardness, as it was investigated can be even comparable to tool hardness. The reason must be specific structure as result of specific hardening mechanisms. In these paper we proposed such mechanisms in the case of cold forming of 1H18N9T stainless steel.

2. Experimental procedure

The specimens for the study were buildups obtained in the power spinning process of cup drawn from 1mm thick 1H18N9T stainless steel sheet-metal and the tool made from NC6 tool steel. Experimental setup shows figure 1, where: 1-buildup, 2-tool, 3-cup, 4-core, 5-centre, 6-plane of *perpendicular cut* TEM specimen, 7- plane of *parallel cut* TEM specimen, d- view direction of figure 2. Process was performed with standard turning machine. The buildups were produced for rotational speed equal 22 and 35,5s^{-1} and constant feed per revolution 0.126 mm. As a first the samples for metallography investigations were prepared. In this case the conventional grinding and polishing procedure was used.

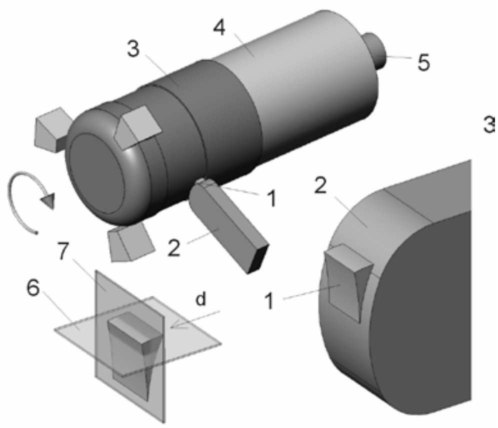

Fig. 1. Experimental setup

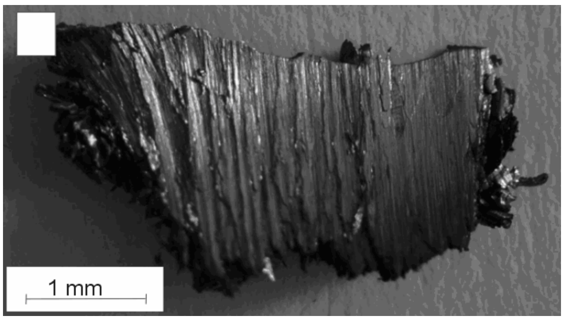

Fig. 2. Removed buildup

For TEM study thin foils method was used. First, out of the removed buildups, figure 2, the rods *parallel* and *perpendicular* with respect to the material movement direction were cut with electro discharge method (EDM). Then, the rods were included in 3mm diameter thin wall tubes using special two component Gatan G1 resin. Next, the discs thickness of 0.1mm were sliced from the rod using load-less wire saw type IF – 07A. Finally these discs were ion mailed with Gatan equipment. Thin foils were observed in Philips EM transmission electron microscope working at 100kV accelerate voltage.

3. Results

Figure 3 illustrates the electron micrographs of the structure in the *parallel* cross-section. In figure 3a the overall structure observed at small magnification is show. It is visible that the grains are elongated and very fine.

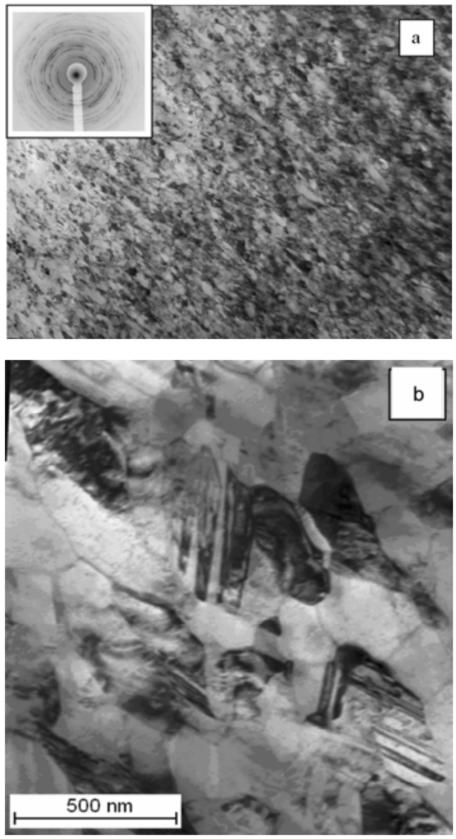

Fig. 3. The structure of buildup in the longitudinal direction: a – electron micrograph showing ultra-fine grains in buildup (x12.000, b – electron micrograph showing micro-twins inside very small grains (x60.000)

As it is documented with selected area diffraction pattern (SAD) located at upper left of figure 3a, the grains are of different orientation, however, as follows from other SAD, but some orientation is preferred. The next photos (figure 3b) illustrate the same structure but in much higher magnification. As looks from this micrograph many grains consists of micro-twins, which are very well visible since their specific bright – dark contrast. In figure 4 the structure of buildups in plane perpendicular to that showed in figure 3 was illustrated. In this case the structure is very "mottled" and complicated. It is characterized with very elongated grains, some of which are bend and oriented perpendicular each to other (figure 4b). More deep observations taken at higher magnification discovered some details which were shoved in figure 5, which illustrates the magnified rectangular area marked in figure 4a. Special attention should be given to the specific lenticular shape of the grain visible in lower part of this micrograph. Inside this grain very fine laths, thickness of approximately 10nm might be identified. (A bit thicker twins formations have been also reported in hydrostatic extrusion of austenitic 316LVM steel under very high deformation [7]). These kind of very thin laths which are nothing else like micro - twins which are often observed in martensite [8]. So we can't exclude that these laths are simply the "super-micro-twins" caused by very high plastic deformation accompanying the process of buildup forming.

The above statement would explain very high hardness of buildups which as measured reached even 510μHV (50HRC). As this statement need to be proved this was given in the next part of these elaboration.

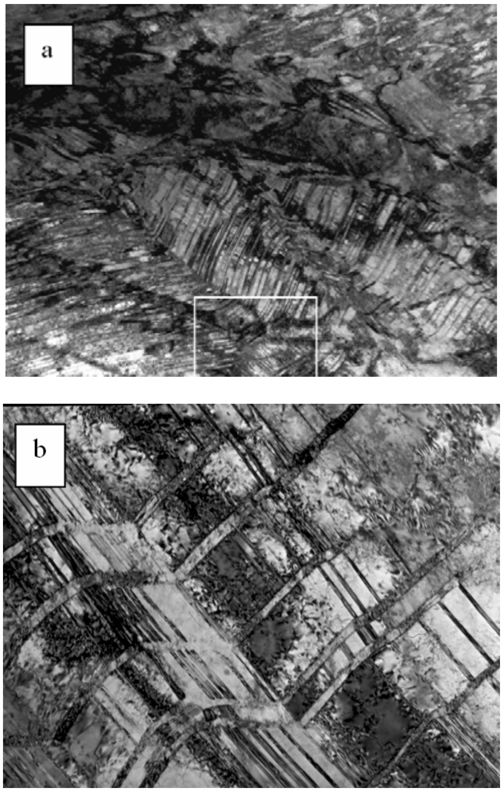

Fig.4. The buildup structure in transverse direction: a - electron micrograph (x12.000), b – electron micrograph of the another place taken at higher magnification (x35.000)

4. Discussion

Very high hardness of buildups forming during different technological processes is a fact and really don't need to be documented. Many authors interpret this as a result of very small grain size [9,10,11], which according to Hall – Petch's relationship [12] lead to hardening caused by grain boundary dislocation blocking. There is no question that increase of dislocation density [13] and grain boundary effect is qualitatively correct, however nobody proved it qualitatively.

The authors are not going to do this in this work also, but we would like to discuss some effect which could be considered as an extra hardening component.

As we already underlined discussing the structure showed in figure 5a, the lath observed are very similar like micro-twins formed in martensite.

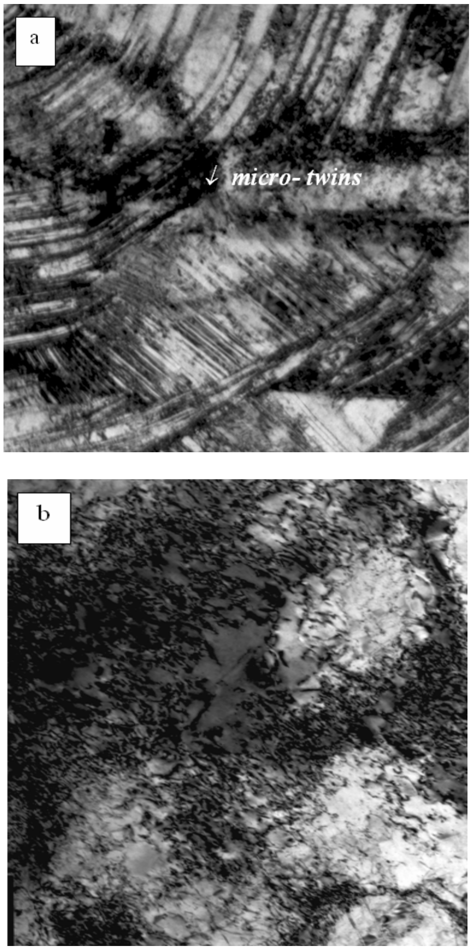

Fig.5. TEM micrograph of build up structure: a – the high magnification of micro - twins showed in fig.4a (total magn. x65.000), b – the region of showing high density dislocation structure in 1H18N9T stainless steel (x40.000), c – electron diffraction pattern with arrows showing the rings from martensite

Now, we return to electron diffraction pattern which all can be indexed for FCC structure. However, apart from reflex from FCC lattice, some extra, usually much weak diffraction rings, were identified. The results of measurements the diameter of the diffraction rings and its analysis was given in table 1, where bold letters were used for weak reflex identified on electron diffraction patterns.

In the third column the ratios of diameter of "i–th" ring to the diameter of the strongest denoted as D_1 were inserted and in the next the Miller indices for FCC lattice. It is visible that there are empty places for some ratios which don't fix to the FCC lattice [14]. In the next step the microscope constant 2λL was evaluated using 5 rings for which interplanar distances for FCC lattice were attributed (table 1). Having microscope constant 2λL the interplanar distances for unknown reflexes were calculated and inserted in column 5-th. These are practically the same as those given in next column, where the interplanar distances for BCC lattice are supported [15]. So it is reasonable to conclude, that the extra reflexes identified in the electron diffraction patterns results from carbonless martensite produced by shearing accompanying the process of deformation.

Table 1.
The results of diffraction pattern analysis

Reflex No.	Ring diam. D_i	D_i / D_1	(hkl) FCC	d_{calcul}	d_{ASTM}^{BCC}	(hkl) BCC
	[mm]	-	-	[nm]	[nm]	
1	34.0	1.00	111			
2	39.2	1.153	200			
3	49.0	1.441	-	0.1438	0.1433	002
4	55.8	1.641	220			
5	60.4	1.776	-	0.1167	0.1170	112
6	65.2	1.918	311			
7	68.2	2.006	222			
8	78.0	2.294	-	0.0903	0.0906	013
9	85.9	2.526	331			
10	88.0	2.588	420			
11	92.6	2.723	-	0.0761	0.0766	123
12	96.4	2.835	422			

Now we would like propose that enormous high hardness of stainless steel is caused by three mechanisms. The first is grain refinement (figure 3a) which leads to formation many grain boundaries playing effective role as obstacles for dislocation movement. The grain refinement includes not only the grain size decrease but also abundant twinning (figure 3b), which supplies extra coherent grain boundaries. Either, ordinary and twin boundaries, causes hardening according to Hall-Petch relationship. The next is strain hardening caused by increase of dislocation density (figure 5b). These dislocations form high density tangles working as effective obstructions for free dislocation movement. Finally, on the basis both microscopy observations and electron diffraction patterns analysis we propose that formation of martensite is one more reason for stainless steel hardening. The martensite formation might be caused by very high plastic strain leading to shearing which is known as a mechanism operating in high manganese Hadfield steel. Formation of martensite was proved by careful analysis of many electron diffraction patterns, where extra reflexes were identified and as follows from calculation, these very well fix to BCC lattice. The reason for lack of tetragonality (c/a) typical for carbon steel is that the forming martensite is carbonless. All of this leads to enormous high hardness of rather soft stainless steel reaching the level 510μHV (50HRC).

5. Conclusions

According to TEM observations and above discussion we propose the following mechanisms responsible for hardening of buildups formed in 1H18N9T stainless steel. These are:

- hardening caused by grain refinement which consists many twins,.
- strain hardening leading to high density of dislocation structure,
- formation of carbonless martensite whose appearance usually is accompanied with high elastic strains.

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