WEAR MECHANISMS OF COATED STEELS DURING PRESS HARDENING

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Abstract

In this work microstructure of Al-base coated press-hardening steel 22MnB5, austenitized at 920°C, was analyzed with respect to the phase formation at the steel/coating interface. Thereby, iron diffusion into the Al-base coating led to the formation of intermetallic Al_xFe_y phases. The intermetallic phases transformed to more Fe-rich phases with increasing dwell time. The coating was characterized by a layered structure of phases Al_5Fe_2 and AlFe after a dwell time of t_{Aus} = 6 min (conventional press-hardening parameters). Cracks were formed in the coating due to the low fracture toughness of the formed intermetallic phases mainly of type Al13Fe4, Al5Fe2 and AlFe. Forming and quenching in the press-hardening tools took place at a temperature range of approx. 800°C to room temperature (RT). We found that the high hardness (520-1130 HV) and low fracture toughness of the intermetallic phases led to strong wear of the press-hardening hot-work tool steel X38CrMoV5-3. The hardness of the intermetallic phases decreased in the direction of a higher temperature, leading to less abrasive wear at higher forming temperatures, thus wear by adhesion is more pronounced. Phase AlFe was found to mainly induce adhesive wear, while phase Al_5Fe_2 led to strong abrasive wear due to the delamination of coating particles.

Keywords: Al-base coating, Intermetallic phases, Hardness, Wear, Press-hardening, Temperature

Introduction

Security relevant structures in automotive bodyworks are conventionally manufactured using high strength steels which possess a high strength to density ratio. Component parts such as longitudinal rear rails, B-pillar reinforcements and inner roof rails are produced using the high strength steel 22MnB5 [1,2]. The steel 22MnB5 is processed by direct press-hardening due to its low formability in hardened condition. During direct press-hardening, steel sheets are austenitized at temperatures of 880-950°C. Subsequently, the steel sheets are transported into the forming tool and then formed and quenched in one step [3]. After cooling, the formed steel sheets have a fully martensitic microstructure and a strength of 1500 MPa [4]. A protective Al-base coating is commonly deposited on the steel surface prior to the press hardening process, thus preventing strong oxidation of the steel sheets during austenitization. Thereby, high austenitization temperatures (880-950°C) lead to diffusion processes at the steel/Al-coating interface and to the formation of intermetallic Al_xFe_y phases in the coating [5,6].

Wear of the press hardening tools strongly depends on the mechanical properties (hardness, fracture toughness) of the Al-base coating during cooling and quenching. The mechanical properties of the Al_xFe_y intermetallics were only fragmentary investigated by Köster et al. and Kobayashi et al. [7,8]. They found that especially Al-rich Al_xFe_y intermetallics of types of $Al_{13}Fe_4$ and Al_5Fe_2 exhibited high hardness and low fracture toughness, simoultaneously. In this context, low fracture toughness promotes crack formation and propagation in the Al-base coating microstructure which may lead to the delamination of coating constituents and thus can cause

wear of the press hardening tools. However, mechanical properties of the intermetallic phase were only measured at room temperature. Mechanical properties at higher temperatures of the forming and quenching process (800°C to RT) were not published.
This work deals with the investigation of the phase formation in the Al-base coating of steel 22MnB5 during press-hardening. The hardness of the formed intermetallic phases was measured at different temperatures according to the forming and quenching process. Furthermore, the wear behavior of the press-hardening hot–work tool steel X38CrMoV5-3 was investigated with respect the formed intermetallic Al_xFe_y phases at different temperatures.

1. Experimental

Materials and heat treatment

Steel sheets of 22MnB5 with a thickness of 1.5 mm were coated with an AlSi10Fe3 layer (17 to 25 µm) by hot dipping and manufactured industrially by Thyssen Krupp Steel in Duisburg (Germany). The chemical composition of steel sheets of 22MnB5 and the AlSi10Fe3 coating is given in ***Table 1***.

Table 1: Chemical composition of the materials used [mass%]; measured by spark spectrometry (22MnB5) and EDS (AlSi10Fe3) in hot-dipped condition [5]

	C	Si	Al	Mn	Cr	Ti	B	Fe
22MnB5	0.234	0.289	0.034	1.258	0.119	0.028	0.002	bal.
AlSi10Fe3		10.23	bal.					2.16

Squared specimens (20 x 20 mm) of the Al-base coated 22MnB5 sheets were heat-treated in argon atmosphere at T_{AUS}= 920 for t_{Aus} = 1, 2 and 6 min. The cross-section of each specimen was ground with 18 µm abrasive paper and polished with a 3 µm diamond paste and ¼ µm SiO_2 polishing suspension. Microstructural examinations were performed by means of scanning electron microscopy (SEM) using device Leo-Gemini 1530VP at a working distance of 12 mm and a current of 21 keV. The chemical composition of the structural constituents was analyzed by energy-dispersive X-ray spectrometry (EDS) using an acceleration voltage of 10 keV and a working distance of 8.5 mm.

Wear specimens of the hot work tool steel X38CrMoV5-3 with a spherical testing surface (r = 5 mm) were hardened and tempered in accordance to the manufacturer’s instructions (T_{AUS}= 1060°C, T_{temp}= 520°C) in an inert gas furnace to a hardness of 630 HV30. Specimens of the intermetallic phases Al_5Fe_2 and AlFe were produced by powder metallurgical route. Aluminum and iron powder was mixed in accordance to the respective stoichiometry. The powder mixture was then fully melted at 1300°C for 1 h under argon atmosphere in a hot press. The intermetallic phases were formed by solidification during slow cooling down to room temperature. The existence of the formed phases was confirmed by EBSD investigations.

Phase Analyzing

Determination of the formed phases in the AlSi10Fe3 coating was performed by diffraction measurements using synchrotron radiation. The measurements took place at the Delta electron storage ring in Dortmund (Germany) using a wavelength of λ= 0.45919 Å. Debye-Scherrer circle segments (140-155°) were integrated and conditioned with the program Fit2D (ESRF). Integrated diffractograms were analyzed using X-Pert analyzing software and ICDD-JCDPS database PDF-2. The local distribution of phases in the coating microstructure was performed by EBSD measurements with a Leo 1530 VP Gemini SEM equipped with an EBSD camera (TSL-DigiView1612). The EBSD measurements were carried out at a working distance of 13 mm with a tilt of 70°, an acceleration voltage of 20 keV, and an extractor voltage of 6.2 keV. The results were conditioned and evaluated using OIM analysis software (TSL).

Hardness measurements and wear experiments

Hardness and wear measurements were performed with an Optimol SRV4 measuring device at room temperature, 400 and 800°C. Hardness measurements were carried out in accordance to DIN EN ISO 6507-1 with an applied load of 4,903 N (HV0.5). Steel X38CrMoV5-3 specimens were tested against the before polished surface of the Al_5Fe_2 and AlFe specimens with a frequency of 10 Hz over a distance of 4 mm (F = 10 N, t = 30 s) in a sliding wear test. Hardness and wear measurements were performed in forming gas to avoid strong oxidation of the worn surfaces.

2. Results and Discussion

In the first section, the phase formation at the steel/coating interface during press-hardening will be presented. Subsequently, the mechanical properties of the formed intermetallic phases and the wear behavior of the hot-work tool steel X38CrMoV5-3 will be discussed.

2.1 Phase formation at the steel/coating interface during press-hardening

Thin layers of intermetallic phases at the steel/coating interface were already formed during hot dipping of the steel sheets in the Al-base melt. On this account, intermetallic phases Al_8Fe_2Si, Al_5Fe_2, and $Al_2Fe_3Si_3$ could be identified after hot dipping (**Fig. 1**). The formation of these phases can be explained by intensive iron diffusion into the liquid aluminum during hot dipping. With increasing iron content in the aluminum melt, the liquidus temperature increases and phase Al_8Fe_2Si was formed as a result of solidification processes. The phase Al_8Fe_2Si transforms to Al_5Fe_2 with increasing iron diffusion into the coating and thus increasing iron content. In addition, $Al_2Fe_3Si_3$ precipitates were formed at the Al_5Fe_2 boundaries, as a result of the low solubility of the element silicon in the phase Al_5Fe_2. The phase formation during hot dipping and the following press hardening process was analyzed in detail in [5].

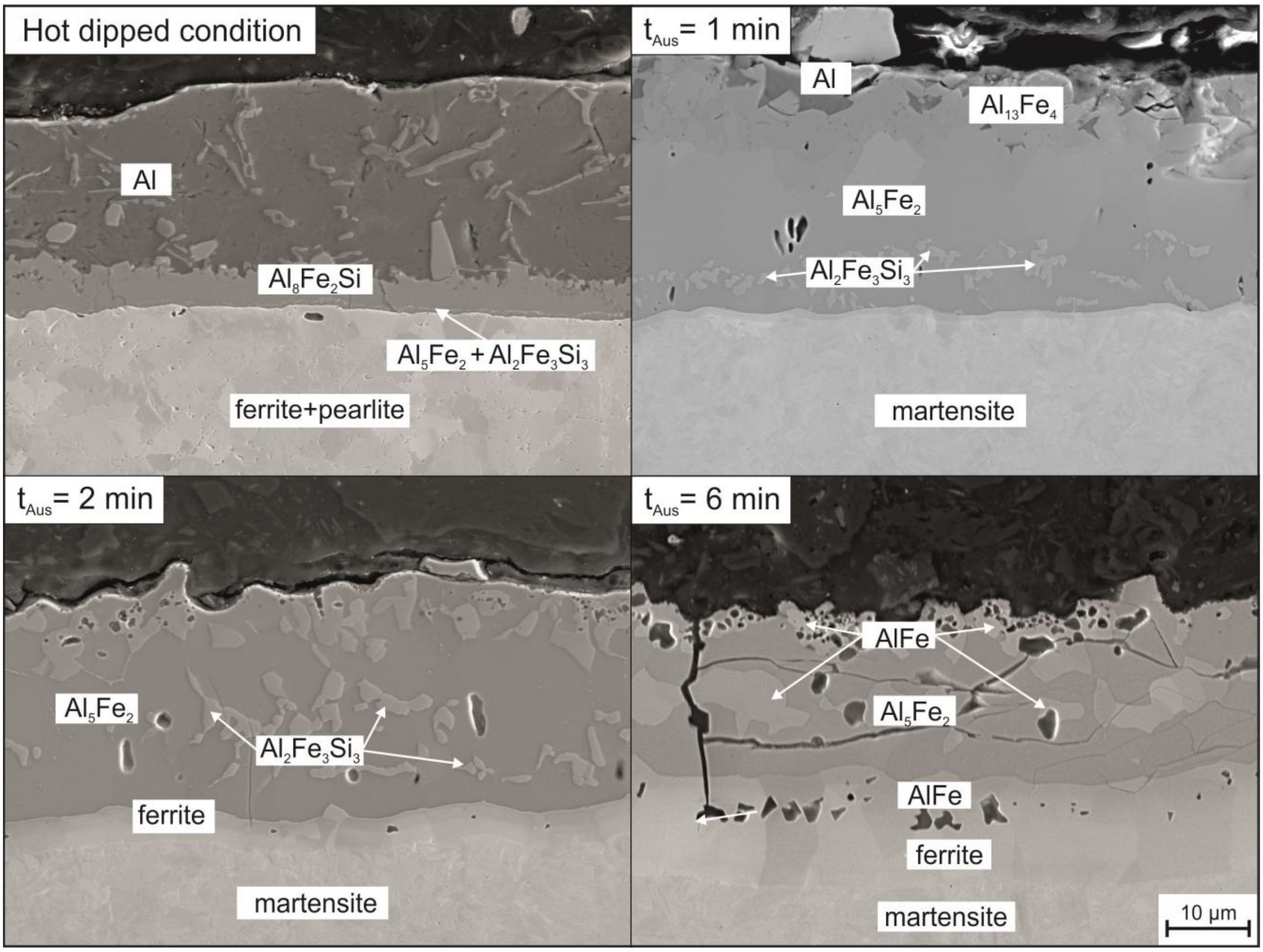

Fig. 1: SEM micrograph of the phase formation in the AlSi10Fe3 coating after hot dipping and austenitization for different dwell times at 920 °C.

The coated steel sheets are austenized at 920°C for a dwell time of 6 min in the conventional press-hardening process. During the first minute, strong diffusion of iron into the Al-base coating led to the growth of phases $Al_{13}Fe_4$ and Al_5Fe_2 in the direction of the coating. As indicated by **Fig. 1**, the growth of the Al_5Fe_2 phase was stronger compared to that of phase $Al_{13}Fe_4$. The faster formation of the phase Al_5Fe_2 can be explained by the high vacancy rate of 30% along the c axis in the orthorhombic Al_5Fe_2 lattice [9]. The high vacancy rate supports directional diffusion of iron towards the Al_5Fe_2 phase in the Al-base coating. The precipitations of type $Al_3Fe_2Si_2$ grew at the grain boundaries in the phase of type Al_5Fe_2. After two minutes, the Al-base coating was completely transformed into the intermetallic phases Al_5Fe_2 and $Al_2Fe_3Si_3$ (**Fig. 1**). The steel substrate was strongly enriched with aluminum near the steel/coating interface. Aluminum stabilizes the bcc lattice of iron. Thus, the austenitic microstructure transforms into α-Fe with increasing aluminum diffusion into the steel substrate during austenitization at 920°C. The formed α-Fe layer did not transform into martensite during the following quenching. Thus, a thin ferrite layer remained at the steel/coating interface after the press hardening process. The thickness of the formed α-Fe layer increased with increasing dwell time. Formation of $Al_xFe_ySi_z$ intermetallics in the steel substrate was not detected. The different diffusivity of Al and Fe in the intermetallic phases led to the formation and growth of Kirkendall pores at the steel/coating interface. The size of the Kirkendall pores was also increased with increasing dwell time, which can be traced back to the opposed diffusion of the elements Al and Fe at the steel-coating interface.

Between a dwell time of 2 and 6 min, the phases Al_5Fe_2 and $Al_2Fe_3Si_3$ transformed into the more Fe-rich phase AlFe. Silicon was solved in the phase AlFe (~12 at.-% [5]) and formed silicon-rich oxides on the coating surface. After a dwell time of t_{Aus} = 6 min, the coating consisted of a layered structure of the phases Al_5Fe_2 and AlFe. Furthermore, high crack density could be identified in the coating. After a dwell time of t_{Aus} = 6 min, the austenized steel sheets are conventionally transported into the forming tool to be quenched and formed. During this process, the forming tools are in direct contact to the coating surface.

2.2 Wear mechanisms during press hardening

The hot-work tool steel X38CrMoV5-3 possesses a hardness of approx. 630 HV in quenched and tempered state. The hardness of phase Al_5Fe_2 was found to be much higher (1130 HV), while the hardness of phase AlFe (520 HV) was below the hardness of the hot-work tool steel at room temperature. Thus, especially phase Al_5Fe_2 should lead to strong abrasive wear of the tools during forming and quenching at lower temperatures. The low fracture toughness promotes the delamination of coating particles and further increases the abrasive wear of the tools.

Table 2: Hardness and fracture toughness of the phases Al_5Fe_2 and AlFe [5,10]

	Hardness [HV0.1]	Fracture Toughness [$MPa/m^{1/2}$]
AlFe	520	26
Al_5Fe_2	1130	~1

However, the presented results were measured at room temperature. During the quenching and forming process, the coated steel sheets and thus the intermetallic phases undergo a temperature profile from approx. 800°C (after transport from the furnace into the tool) to room temperature. Consequently, the wear mechanisms of the hot-work tool steels during quenching and forming depend on the mechanical properties of the intermetallic phases at the respective forming temperatures. The hardness of the phases AlFe and Al_5Fe_2 at RT, 400°C and 800°C are presented in **Fig. 2**. In contrast, room temperature hardness of tool steel X38CrMoV5-3 is indicated as a dashed line.

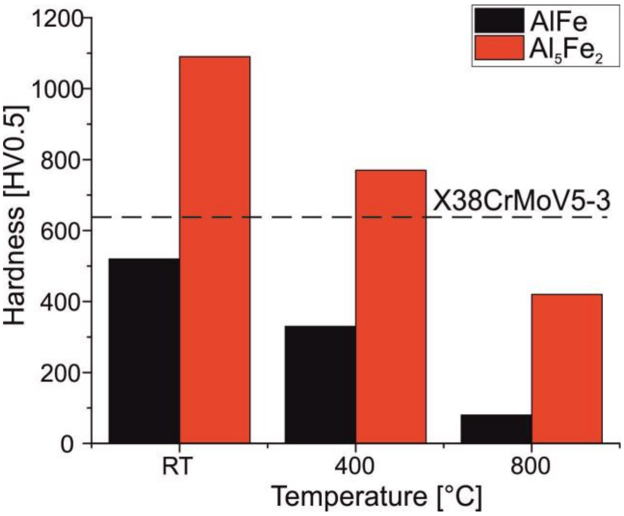

Fig. 2: Hardness of the intermetallic phases Al_5Fe_2 and AlFe at different temperatures

The hardness of both intermetallic phases decreased with increasing temperature. The hardness of phase AlFe was lower than the hardness of the hot-work tool steel at each temperature. A minimal hardness of 80 HV was measured at 800°C. The hardness of phase Al_5Fe_2 was lower than the hardness of the hot-work tool steel at 800°C, but increases above this hardness at lower temperature of 400°C and RT. Thus, the tool wear should be lower during cooling and forming from 800°C to 400°C than subsequently during cooling from 400°C to RT.

Wear experiments of the hot-work tool steel X38CrMoV5-3 against specimens of the phases Al_5Fe_2 and AlFe were performed to evaluate the temperature-dependent wear mechanisms. **Fig. 3** shows the worn surface of the hot-work tool steel after wear experiments against AlFe and Al_5Fe_2 at 400°C and 800°C.

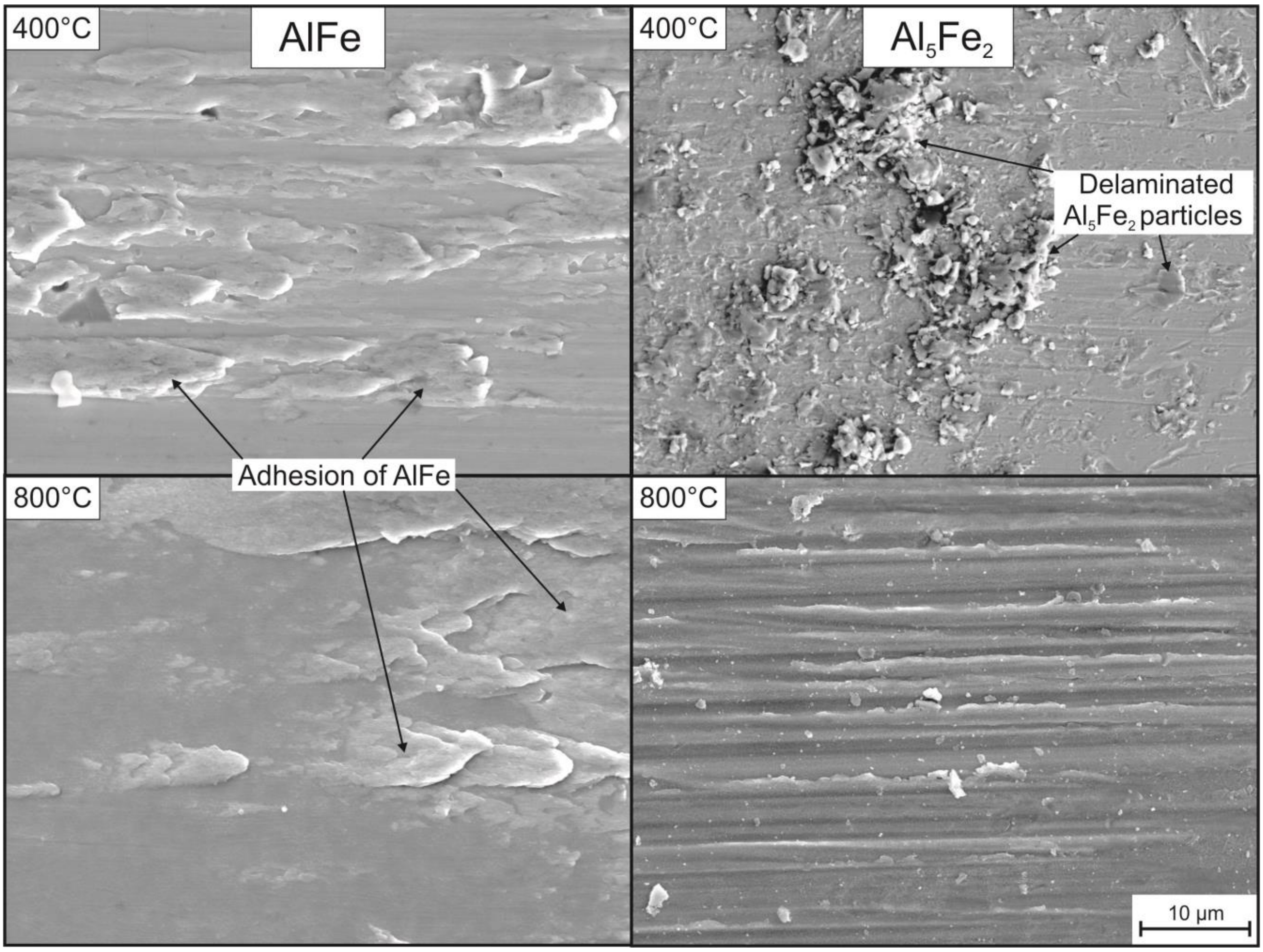

Fig. 3: SEM micrographs of the X38CrMoV5-3 hot-work tool steel worn surfaces after wear tests against AlFe and Al_5Fe_2 at 400°C and 800°C.

Fig. 3 shows that the phase AlFe mainly led to adhesive wear on the tool steel surface. Locally, welded particles of phase AlFe could be identified. The wear mechanism and intensity were almost similar for both investigated temperatures. Phase Al_5Fe_2 led to strong abrasive wear on the steel surface at 400°C, due to the delamination of brittle intermetallic particles. The delamination of intermetallic particles can be explained by the low fracture toughness of Al_5Fe_2. These particles remained on the tool steel surface and caused additional wear. Delamination of brittle intermetallic particles could only be locally identified on the steel surface after the wear experiment at 800°C. Thus, the fracture toughness of phase Al_5Fe_2 seemed to be strongly increased at this temperature. Investigations of the steel surface structure after the wear test at 800°C indicates that adhesive and abrasive wear occurred. Locally, melted aluminum as well as delaminated Al_5Fe_2 particles could be identified on the steel surface.

Summarized, wear mechanism on the hot-work tool steel surface can be described by the micro-mechanisms of adhesion and abrasion. Thereby, phase of type AlFe mainly led to adhesive wear. In contrast, phase Al_5Fe_2 promoted abrasive wear due its high hardness and the delamination of brittle intermetallic particles. While mainly adhesive wear occurred at 800°C, the amount of abrasive wear increased at lower temperatures.

3. Conclusions

The following key results were found within this work:

- During austenitization, diffusion of iron into the AlSi10Fe3 coating lead to the formation of $Al_xFe_ySi_z$ intermetallics in the coating.
- After two minutes, Al-base coating completely transforms into intermetallic $Al_xFe_ySi_z$ phases.
- The coating consists of a layered structure of phases Al_5Fe_2 and AlFe after t_{Aus} = 6 min (conventional press-hardening parameter). Furthermore, a high crack-density is formed due to the low fracture toughness of the intermetallic phases.
- The hardness of phases AlFe and Al_5Fe_2 decreases in the direction of higher temperatures. The hardness of phase AlFe is below the hardness of the hot-work tool steel X38CrMoV5-3 at all investigated temperatures. The phase Al_5Fe_2 possesses higher hardness than the hot-work tool steel from RT to 400°C. The hardness decreases below the hardness of the tool steel above 400°C.
- Phase Al_5Fe_2 mainly lead to abrasive wear on the tool steel surface, while phase AlFe promotes adhesive wear.

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References

[1] T. Manzenreiter, M. Rosner, T. Kurz, G. Brugger, R. Kelsch, D. Hartmann, A. Sommer, BHM, 157 (2012), pp. 97-101

[2] D.W. Fan, B.C. De Cooman, Steel Research Int., 83 (2012), pp. 412-433

[3] A. Barcellona, D. Palmeri, Metall. Mater. Trans., A, 40 (2009), pp. 1160-1174

[4] H. Karbasian, A.E. Tekkaya, Journal of Materials Processing Technology 210 (2010), pp. 2103-2118

[5] M. Windmann, A. Röttger, W. Theisen, Surf. Coat. Technol. 226 (2013), pp. 130-139

[6] M. Windmann, A. Röttger, W. Theisen, Surf. Coat. Technol. 246 (2014), pp. 17-25

[7] U. Köster, W. Liu, H. Liebertz, M. Michel, Journal of Non-Crystalline Solids 153/154 (1993), pp. 446-452

[8] S. Kobayashi, T. Yakou, Mater. Sci. Eng., A, 338 (2002), pp. 44-53

[9] T. Heumann, S. Dittrich, Z. Met. 50 (1959), p. 617

[10] A. Kubošová, M. Karlík, P. Haušild, J. Prahl, Mater. Sci. Forum 567–568 (2008), pp. 349