



Response to thermal cycling of CAPVD (Al,Cr)N-coated hot work tool steel

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ABSTRACT

The principle failure mechanism in thixoforming dies is thermal fatigue as the mechanical loading on the tooling is modest owing to a mushy feedstock. X32CrMoV33 hot work tool steel samples coated with (Al,Cr)N via Cathodic Arc Physical Vapour Deposition were submitted to thermal cycling under conditions which approximate thixoforming of steels. The PVD AlCrN coating provides adequate protection against oxidation of the hot work tool samples, shown to be one of the predominant mechanisms leading to thermal fatigue cracking. However, it does not last very long due to the extensive softening of the tool steel substrate and the thermal expansion mismatch between the hot work tool steel and the nitride coating.

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1. Introduction

Tooling is a significant issue in steel thixoforming [1]. The major causes of thixoforming die failure were reported to be thermal fatigue, wear and oxidation [2–5]. Thermal fatigue results from cyclic heating and cooling of the die surface during the forming process. The abrasion and impact of the solid α -Fe globules of the mushy feedstock leads to substantial wear while oxidation of tool surfaces is inevitable at the thixoforming temperature range. Oxidation and wear have a synergistic effect, creating more damage together than if either were acting alone [6]. Oxidation has a negative impact also on the thermal fatigue performance [2].

There appears to be two major routes to combat the hostile thixoforming environment. One is to use materials better suited for high temperature use [7–9]. Potential materials must exhibit a superior wear resistance, capacity to retain mechanical strength at elevated temperatures and indeed a much better oxidation resistance. The second and more attractive method is to employ surface engineering techniques on the conventional hot work tool steels, which, without any protection, were shown to be entirely inadequate for such harsh conditions [10]. Of the several advanced surface engineering techniques available to the tool shop, Physical Vapour Deposition (PVD) provides superior wear and high temperature resistance and is capable of depositing ceramics below the temper softening temperature of tool steels [1,11,12]. Recently, AlCrN coatings have received much attention owing to their excellent properties particularly at elevated temperatures [13]. While AlCrN hard coatings have

been successfully applied as protective layers for stamping and forging tools [14–26], there have been very few attempts to explore the potential of PVD AlCrN coatings for thixoforming tools in semi-solid processing of steels [27]. The present work was undertaken to investigate the potential of an AlCrN coating with 42.9 at.% Al, 27.6 at.% Cr and 29.5 at.% N, deposited on X32CrMoV33 hot work tool steel via Cathodic Arc Physical Vapour Deposition (CAPVD) process for steel thixoforming conditions.

2. Experimental

The substrate, X32CrMoV33 hot work tool steel (Table 1), was austenitized at 1025 °C for 30 min, quenched in circulating air and finally tempered twice at 625 °C for 2 h yielding a hardness of 45 HRC. 70/30 at.% Al–Cr cathodes were used to deposit AlCrN coatings on tool steel with the CAPVD process with an industrial size cathodic arc unit. The chamber was evacuated to approximately 10^{-3} Pa. The samples were ion etched with chromium ion bombardment and were heated up to 350 °C before the deposition step which lasted for 75 min. DC-substrate bias voltage was –150 V. The thickness of the coatings was determined using a ball cratering unit. Ultra micro hardness tester was employed to measure the coating hardness. The coating morphology and composition were determined using a JEOL 6335F model field emission gun scanning electron microscope (FEG-SEM) fitted with an Oxford INCA model energy dispersive X-ray analyzer (EDS). The structure of the coatings was examined by a PANalytical X'pert Pro model glancing incidence X-ray diffractometer, with a high resolution ψ goniometer. Cu K α radiation at an incidence angle of 2° was employed to identify the phases in the coatings.

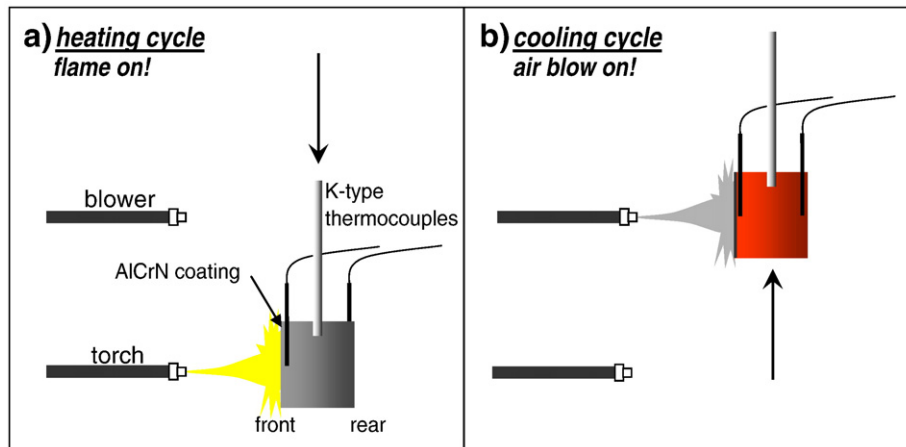
Prismatic coated tool steel samples (25mm × 25mm × 20mm) were cycled between 750 °C and 450 °C, the maximum and the minimum

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Table 1

Chemical composition of the X32CrMoV33 hot work tool steel, measured with an optical emission spectrometer (wt.%). Average of three measurements are listed.

C	Si	Mn	Cr	Mo	Ni	Al	Cu	Nb	V	W	Fe
0.281	0.190	0.200	3.005	2.788	0.221	0.025	0.165	0.002	0.413	0.020	92.63

**Fig. 1.** Experimental set up for the thermal fatigue test; (a) heating and (b) cooling cycles.

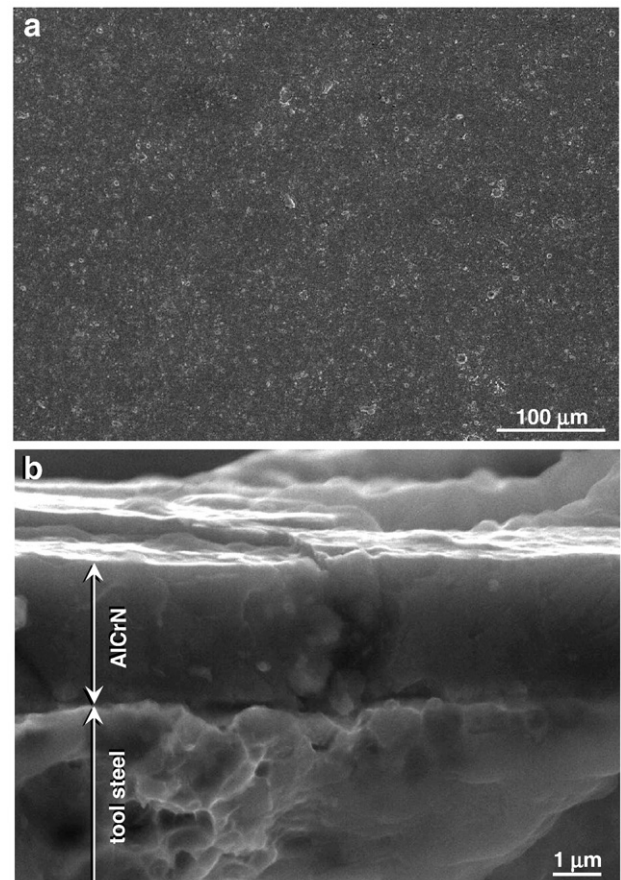
temperatures the die cavity surface had to endure in steel thixoforming experiments (Fig. 1). Both heating and cooling were employed from the coated front face to set up thermal gradients similar to those which prevail in the thixoforming process. These tests are described in detail elsewhere [2] and were repeated in exactly the same fashion with uncoated tool steel samples to identify the impact of AlCrN coatings on the thermal fatigue performance of X32CrMoV33 tool steel. The thermal fatigue tests were conducted until cracks were detected on the surface.

3. Results and discussion

The AlCrN coating reveals uniform features both over the surface and across the section obtained by the fracture of a coated sample (Fig. 2). The average thickness of the coating is $3 \pm 0.1 \mu\text{m}$. Its hardness was measured to be $2600 \pm 50 \text{ HV}$, in reasonable agreement with those reported for AlCrN coatings [28]. Its composition was measured by EDS over an area of 10^{-6} m^2 at three different locations and the average was estimated to be 42.9 at.% Al, 27.6 at.% Cr and 29.5 at.% N. The EDS analysis is not very reliable for light elements such as N and only the Al/Cr is considered suggesting a coating of the type $\text{Al}_{0.61}\text{Cr}_{0.39}\text{N}$. Al/Cr ratio is different from the target composition as some Al is lost. The glancing incidence X-ray diffraction spectrum from the coating is shown in Fig. 3a. The AlCrN coating crystallized in the B1 NaCl crystal structure with a (111) preferential orientation as inferred from a (111) reflection with a measured intensity higher than the standard. The Bragg reflections of the AlCrN coating fall between those of B1 AlN (International Centre of Diffraction Data (ICDD) Card No. 00-046-1200 given in Fig. 3b) and B1 CrN (ICDD Card No. 04-007-9936 given in Fig. 3c), implying complete solubility of Cr, Al and N in the rock-salt-type lattice. The shift in the 2θ values of the (111), (200) and (220) reflections in the AlCrN coating with respect to those of CrN is attributed to a decrease in lattice parameter of the former due to the substitution of some Cr atoms by the smaller Al atoms in the CrN lattice [29]. The cubic, instead of the hexagonal crystal structure of the present coating is linked with its relatively higher Al content and is very welcome. It has been reported that the hardness, oxidation resistance and the tribological properties of $\text{Al}_x\text{Cr}_{1-x}\text{N}$ coatings improve with increasing Al content provided that the cubic structure is retained [22,23]. The oxidation rate is almost

tripled when the hexagonal structure forms at higher ($x > 0.75$) aluminium contents [23].

Typical thermal cycles recorded near the front and rear faces of the AlCrN-coated hot work tool steel are illustrated in Fig. 4a and show

**Fig. 2.** SEM micrographs taken from (a) the surface and (b) the section of the AlCrN coating obtained by the fracture of a coated sample.

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