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Thermal cracking behavior of multi-layer LAFAD coatings on nitrided die steels in liquid aluminum processing

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Abstract

Thermal cracking is the primary mode of failure of large steel dies in aluminum die casting of automotive engine blocks or transmission housing. This cracking is due to the large thermal shock experienced by the die steel when it is quenched by cold water after a die casting cycle. The propagation of cracks depends on the severity of the thermal fatigue cycle (peak and range), that is caused by die surface being in contact alternatively with the liquid melt at 700°C and cold water at room temperature. Previous work at The Ohio State University has shown that single layer hard PVD and CVD coatings do not protect the die steel surface from cracking. On the other hand, they may enhance cracking. This paper presents an interesting multi-layer duplex coating approach with a nitriding H-13 steel substrate and a multi-layer, hard outer film applied by LAFAD technique that prevents reaction with the liquid melt, and alters the thermal fatigue behavior. Results of thermal cycling tests indicate that the multi-layer duplex coating system helps reduce the density of the thermal cracks.

Keywords: Thermal cracking; Die casting; Engine blocks

1. Introduction

Thermal fatigue, popularly known in the industry as heat checking, is caused by the alternate heating and cooling of the die surface. The large thermal gradients put the die surface in compression during heating, and in tension during cooling. This leads to a low-cycle thermal fatigue-induced surface cracking, deterioration of the surface-finish and ultimately die failure. Fig. 1 shows the cracks in a core die insert. Noesen [1] defines thermal fatigue as fracture resulting from the presence of temperature gradients that vary with time in such a manner as to produce cyclic stresses in the surface.

The cracking phenomenon can be divided into two stages:

1. Crack initiation: during the first few cycles, high thermal gradient can result in shock, causing crack initiation. Inclusions and other micro-defects act as preferential sites for crack initiation [2].
2. Crack propagation: once the crack has initiated, the propagation depends entirely on the toughness of the substrate and the thermal stresses imposed. After a number of thermal cycles, the die becomes so soft that the applied thermal stresses are enough to cause plastic deformation. The plastic strains keep on accumulating, resulting in low cycle fatigue cracking.

During previous evaluation campaigns at The Ohio State University, multi-layer and duplex coatings, deposited at Universal Energy Systems (UES) on H-13 steel substrate were observed to provide better corrosion-resistance as compared to commercial coatings and single layer coatings [3,4]. These were therefore chosen for evaluation in a thermal fatigue campaign.

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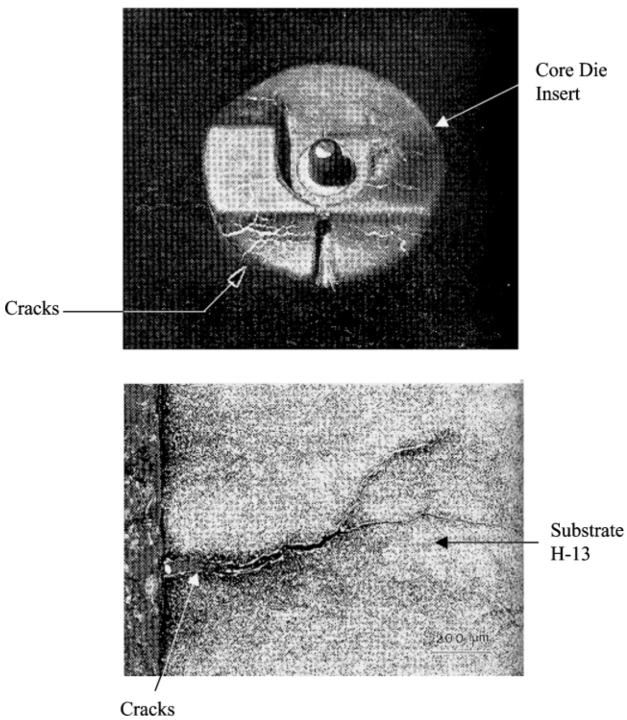

Fig. 1. Cracks in core die insert.

The different coatings tested were a combination of the following systems:

1. Ti/TiC;
2. Ti/TiCN; and
3. Ti/TiAlN.

2. Coating deposition method

During the current study, various multi-layer coatings were deposited on H-13 steel substrates at UES Inc., using a novel 'filtered cathodic-arc deposition' system [5,6]. This large area filtered cathodic arc deposition (LAFAD) technique is described by Gorokhovsky [7] and by Vetter and Perry [8]. In this technique arc sources can be used to extract highly energetic electrons and to ionize the gaseous plasma, such that a 'plasma envelope' that completely surround the part can be created in the coating chamber. Using this technique, very high ion currents can be obtained as compared to the other PVD techniques such as EBPVD and sputtering. The high degree of ionization of the gaseous plasma permits ion saturation levels suitable for ion nitriding. Moreover, when the substrate is strongly biased, significant ion implantation can be achieved.

The arc plasma is generated by a patented electronic trigger and arc-spot control circuitry that effectively eliminates the tendency for the arc spot to be extinguished unpredictably, and provides a stable and continuous operation of the arc for an extended period. The deposition chamber is evacuated to a pressure of 7×10^{-4} Pa prior to the introduction of gases such as argon or nitrogen for cleaning of the substrates or for metal deposition, respectively. The substrates are mounted on a variable speed substrate holder with double-planetary rotation capability, which can be biased to a desired voltage using either a bipolar DC pulse or RF power supply. The test coupons were polished first with emery paper to size 600 grit and then with diamond paste (8000 grit size) before nitriding and coating.

3. Characterization of the coatings

The selected coatings were characterized for thickness, hardness and adhesion. The thickness and layer structures were characterized by Calotest equipment. The total thickness of the multi-layer coating was found to be 4.2 μm. Auger electron microscopy (AES) was used for compositional characterization. Fig. 2 shows the AES depth profile of Ti/TiC, Ti/TiCN and Ti/TiAlN coating candidates.

The hardness of ion-nitrided and coated H-13 coupons was measured using a microindentor with loads of 0.025 and 0.05 kg. Both coated and nitrided H13 steel showed hardness of approximately 11.9 GPa (1220 Vickers) as compared to 5.19 GPa (530 Vickers) for the H-13 steel. Since the coating or ion nitriding was in the range of 3–4 μm, the hardness values represent a composite effect of the coating and the substrate. The surface hardness of the Ti/TiCN multi-layer was measured at 21 GPa (2143 Vickers) and a modulus at 305 GPa. The scratch adhesion tests using CSEM tester indicated that the coatings cracked at loads of 40–60 N.

4. Experiment

The experiments were designed to study the role of nitriding and multi-layer and duplex coatings in curtailing thermal fatigue in die casting dies. The thermal fatigue tests were carried out using the thermal cycle simulator at the Ohio State University [9]. The thermal cycle was obtained by simulating the actual die conditions using a commercial FEA software (DEFORM) [9,10]. Fig. 3 shows the profile of thermal cycle used in our experiments. The test coupon is shown in Fig. 4. The flat surface of the coupon is for observing thermal fatigue cracking whereas the edges show the effect of stress concentration. The coupons were nitrided to two different case depths, namely 150 μm (shallow nitrided) and 350 μm (deep nitrided) to study the effect of case depth on the initiation and propagation of thermal fatigue cracks. The multi-layer coatings evaluated had several layers of Ti/TiC, Ti/TiCN and Ti/TiAlN. In duplex coating, the test coupons (H-13) were first nitrided and then coated with TiAlN. The coupons were subjected to

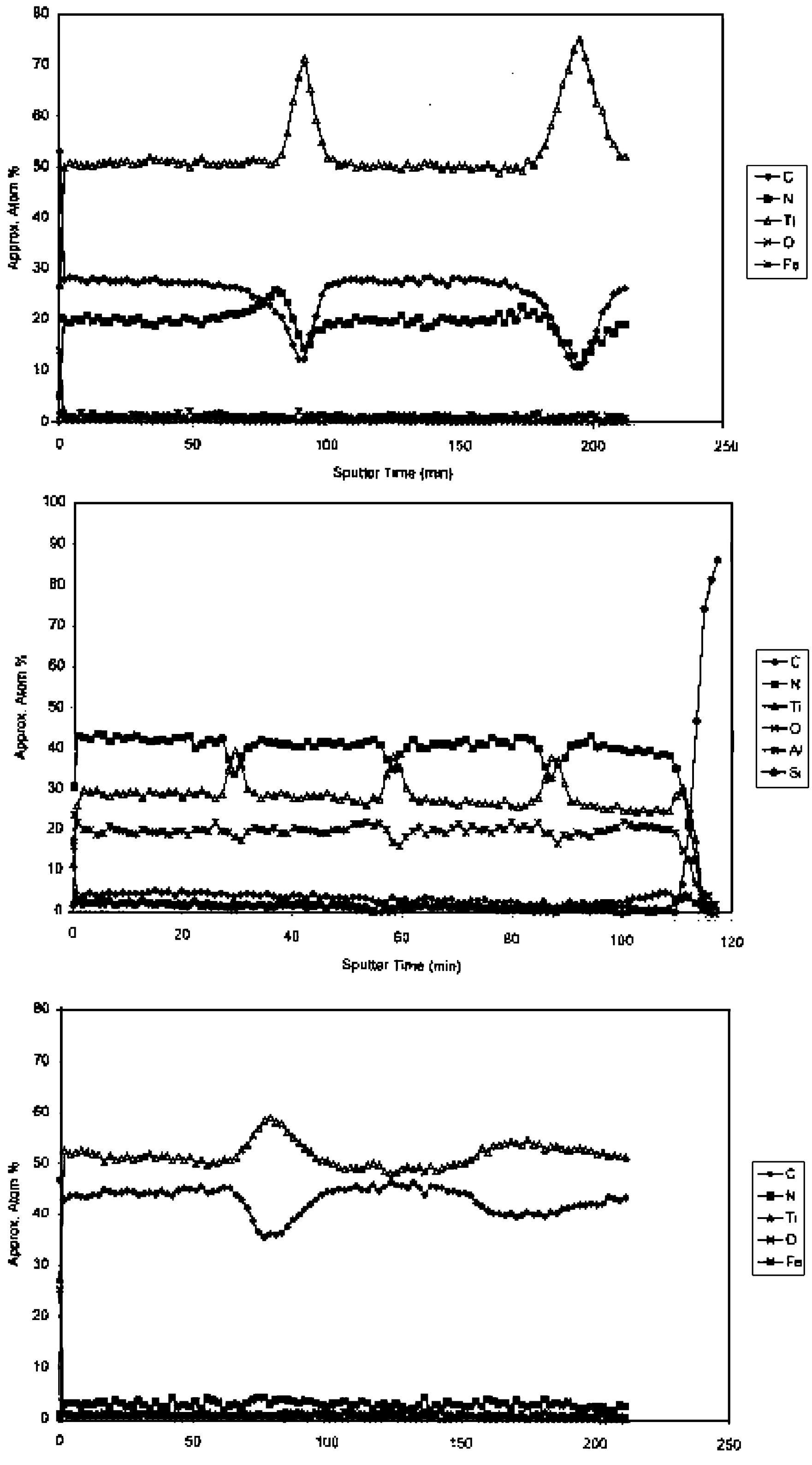

Fig. 2. AES depth profile analysis of Ti/TiCN, Ti/TiAlN and Ti/TiC coatings.

3000 thermal cycles (cleaned with NaOH solution and examined optically after every 1000 cycles).

5. Observations and results

5.1. Results of testing of nitrided coupons (Fig. 5)

After 1000 cycles, in the case of shallow nitrided coupons, the surface was seen to exhibit extensive craze cracking. Wide 'primary' cracks are formed and smaller cracks branch off from these larger cracks. The primary cracks were 15–20 μm wide and had an oxide layer built up inside them. In deep nitriding case also, the cracks exhibited a craze crack network. However, the number of cracks in a given visual field was found to be lower than that for the shallow nitriding case. Unlike the shallow case, the crack boundaries were intact. The cracks were approximately 20 μm wide. Considerable oxide scale was observed in the cracks in the case of un-nitrided coupon.

After 2000 cycles, the crack pattern had not changed noticeably in either case. In the shallow nitrided coupon, most of the small pits along cracks after 1000 cycles seemed to have blended with the cracks due to an increase in width of the cracks. As a result, the crack boundaries were found to be much smoother than those after 1000 cycles. The crack width was, however, found to increase in both the cases. The crack width was found to increase further with oxide accumulation after 3000 cycles.

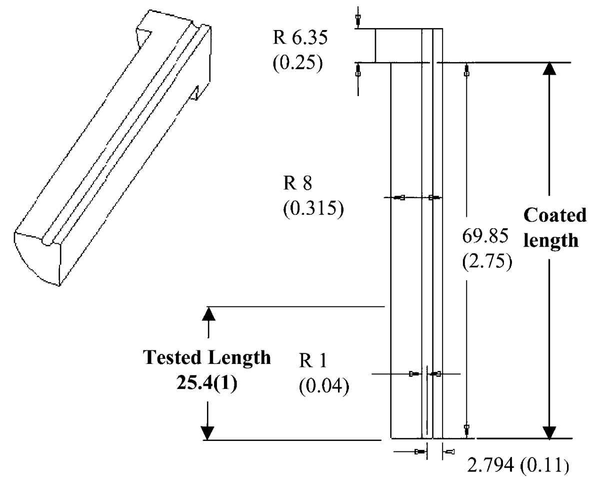

Fig. 4. Test coupon. All dimensions in mm. Dimension in parenthesis are in English units.

The cracks in the case of deep nitriding were found to have propagated farther into the substrate than those in the case of shallow nitriding (Fig. 6).

Fig. 7 shows the microhardness profiles for the shallow and deep case nitriding, before testing and after 3000 cycles. The hardness of both the nitrided samples was seen to reduce from the pre-test values and the

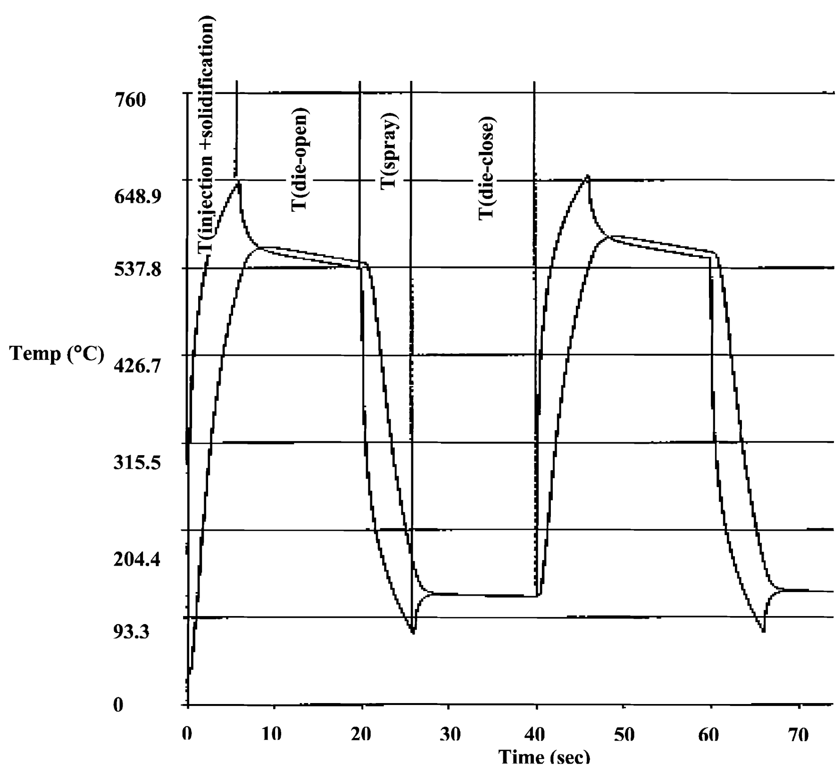

Fig. 3. The time–temperature plot obtained from the DEFORM simulations.

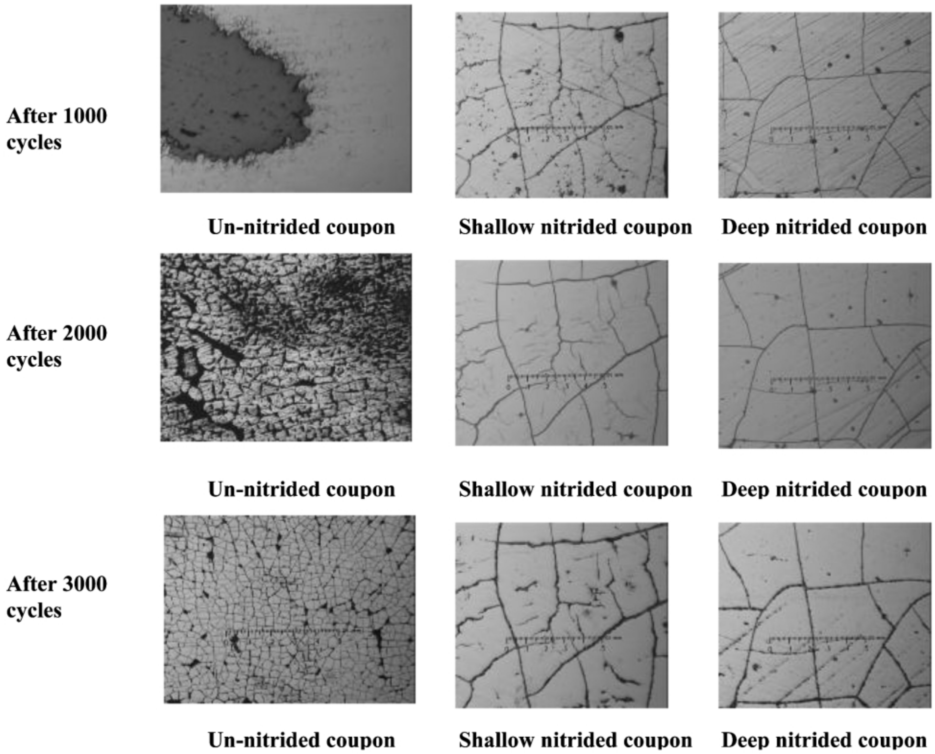

Fig. 5. Results of testing of nitrided H-13 coupons at 50× magnification.

effective case depths were seen to decrease to approximately 50–70 μm. The loss of effective case depth was seen to be higher for the deep nitriding. However, the surface hardness retention was higher in deep nitriding case.

5.2. Discussion

The number of cracks seems to have reduced progressively from un-nitrided coupons to shallow nitrided coupons to deep nitrided coupons; however, the depth of the cracks has shown the opposite trends. In the case of nitrided coupons, the cracks extend up to the case depth, which can be attributed to the high hardness and low toughness of the case. Therefore the crack that is initiated continues to propagate for the full depth of the case till it encounters H-13. The crack also propagates in H-13 but at a much slower rate. The lower number of cracks in the deep nitrided case can be attributed to the higher hardness and yield strength, as this reduces the crack initiation caused by thermal shock. As soon as the crack is initiated, the stresses are relieved, reducing the number of sites for crack initiation.

During these tests, the favorable effect of nitriding on soldering was observed. Soldering was less in nitrided coupons as compared to plain H-13 coupons. The coupon with deep nitrided case was observed to be better than shallow nitrided case in terms of resistance

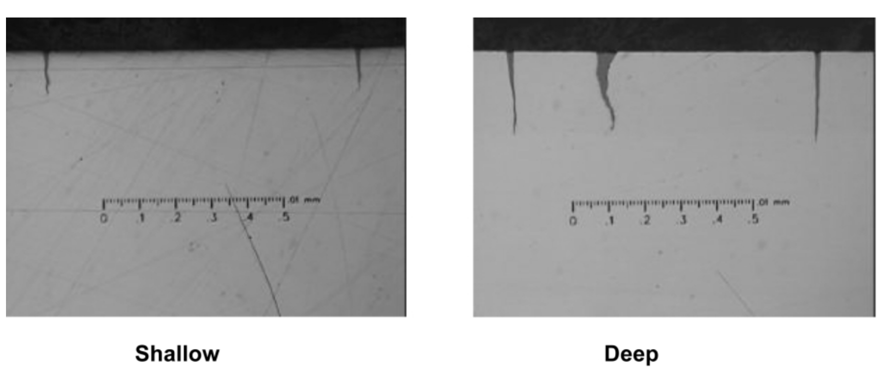

Fig. 6. Micrograph of cracks inside shallow and deep nitrided H-13 coupon after 3000 cycles (100×).

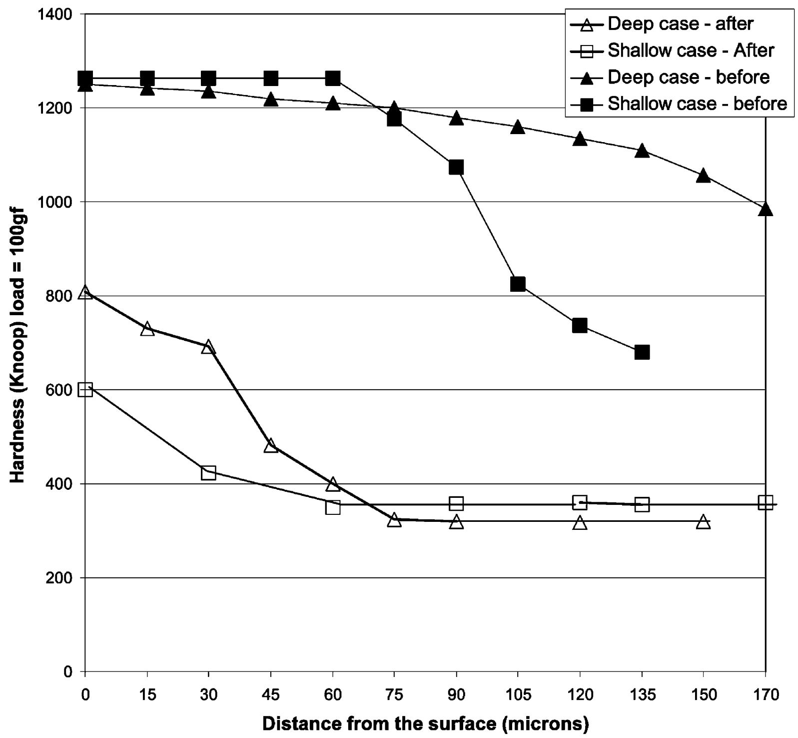

Fig. 7. Microhardness profiles of nitrided samples before and after testing (3000 cycles).

against soldering. This is due to the formation of CrN during nitriding of H-13 (5% Cr). Earlier tests have shown CrN to be good for resistance against soldering.

5.3. Results of testing of multi-layer coated coupons

Fig. 8 shows the results of testing of Ti/TiC, Ti/TiCN and Ti/TiAlN coated coupons. Both Ti/TiC and Ti/TiCN showed almost the same results. A lot of oxide was seen to form on the coated surfaces and small pits, and pores were seen on both coatings. After 1000 cycles, the coatings dissolved at some places while at other places the coatings were seen to have cracked and flaked, leading to soldering and removal of the coating. Some minute cracks, which might have formed in the substrate, were also observed around the pits in both coupons.

In the case of TiAlN coating also, an oxide layer was formed but was thinner than that observed in any of the previous cases. This could be primarily due to rapid oxidation of the TiAlN surface to form hard and impermeable Al_2O_3, which prevented further oxidation. After testing, no crack network was observed on the flat surface after polishing. However, small cracks were seen forming on the edges of the coupon.

5.4. Discussion

The cracking is found to be a substrate driven phenomenon. Once the substrate cracks, it results in the cracking of the coating, leading to flaking and dissolution. The difference between the hardness of the substrate and coating is critical in the removal of coating during early cycling. Therefore, to avoid this, an interface is required between the coating and the substrate that provides a gradual change in hardness. A duplex coating approach with ductile interlayer may work as desired under these conditions.

5.5. Result of duplex coating system

Fig. 9 compares the duplex TiAlN coated coupon with shallow (type 1) and deep nitriding (type 2), before and after thermal cycling. After 1000 cycles, large cracks were seen on the surface of type 1 coupon, which were similar to those seen in the case of uncoated shallow nitriding. The width of the cracks can be estimated to be approximately 15–20 μm. The cracks appeared to have started from the edges and propagated towards the surface. The cracks were seen to be much deeper than the coating thickness of 2.5 μm. The behavior of the type 2 coupons after testing was seen to be similar to that of the uncoated deep nitrided coupon. The cracks were seen to be wider (larger) and fewer than those seen on the type 1 coupon.

5.6. Discussion

The coating was not found to provide any added protection to the nitrided substrate as regards countering

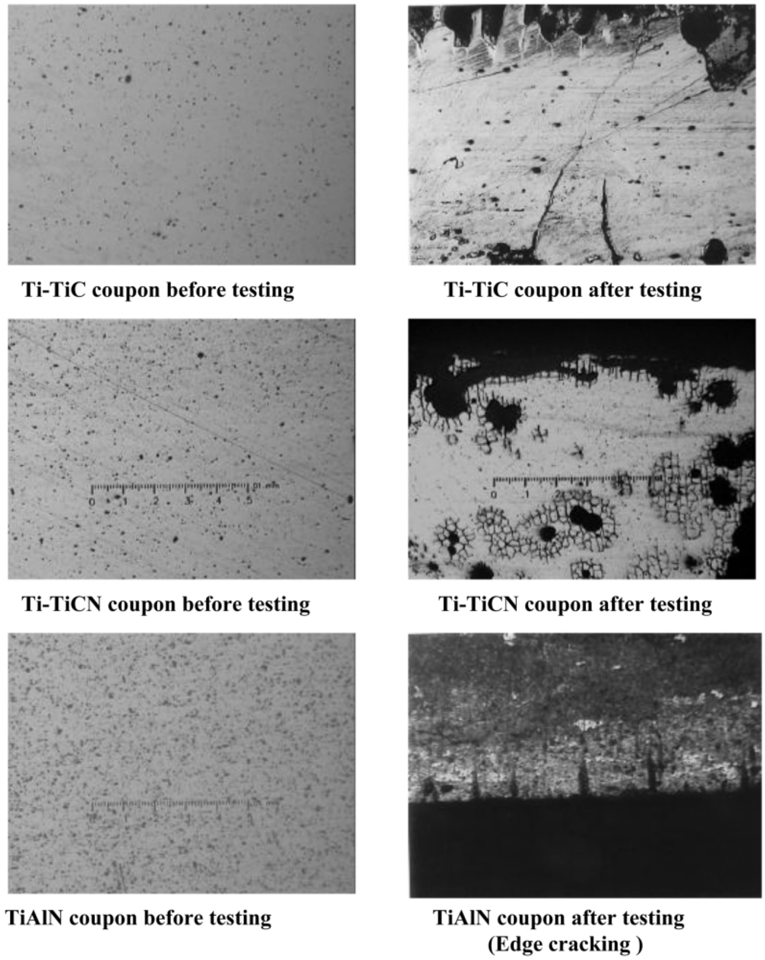

Fig. 8. Coupons with different coatings before and after testing 1000 cycles at 50× magnification.

heat checking behavior except for soldering. In both cases, oxidation and soldering were seen to be less than that seen in the case of pure nitriding, which can be attributed to the formation of Al_2O_3 on the TiAlN surface [11]. Therefore this duplex approach is probably a better approach to counter both heat checking and soldering at the same time.

6. Conclusions

In the present study, multilayer PVD hard coatings were deposited at UES Inc. on plain and nitrided H-13 steel coupons using the LAFAD System. These coatings were evaluated in a thermal cycle simulator using cycles representative of the actual thermal profile in die casting. During the thermal cycling tests, the nitrided coupons were observed to perform better than un-nitrided coupons. The multilayer coating did not prove effective without a good interface. With nitriding, these coatings were found to suppress cracking of the substrate, by delaying crack initiation and reducing the incidence of cracking. However, the depth of cracks has increased. The duplex approach also proved very effective in curbing soldering. This is a significant result, as it demonstrates the beneficial effect of a duplex treatment of die steel for combating a major cause of die failure in the die-casting application. The depth of the crack can also be reduced if a ductile interlayer could be placed between the coating and the substrate. With this duplex approach, the filtered cathodic arc deposition process has demonstrated to be a possible technique to significantly improve the useful lifetime of the die-casting dies.

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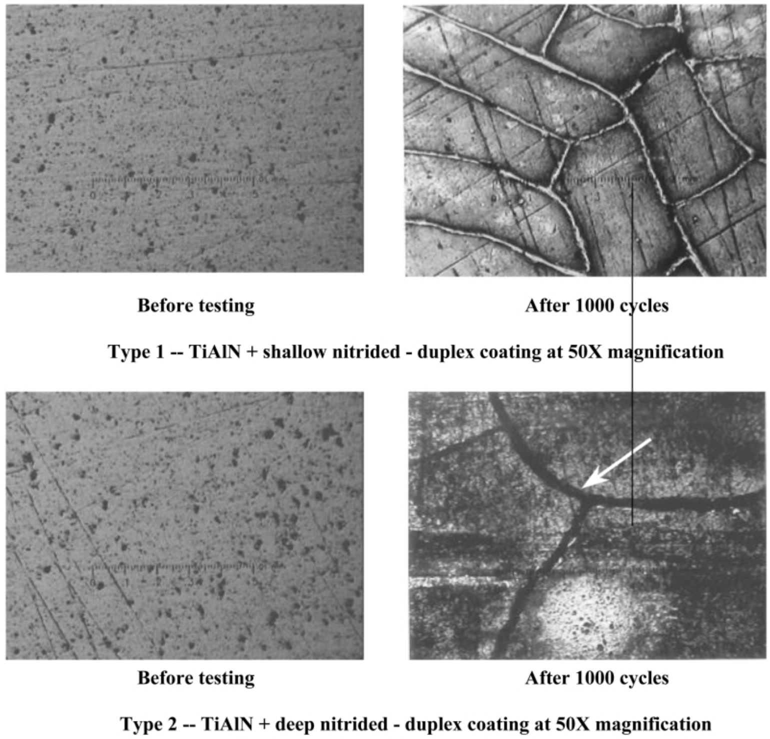

Fig. 9. Result of duplex coating system.

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