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Final Project Report

on

**EVALUATION OF PERMANENT DIE COATINGS TO IMPROVE THE WEAR
RESISTANCE OF DIE CASTING DIES**

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Executive Summary

Die Casting dies are subject to severe service conditions during the die casting operation. While these severe conditions are necessary to achieve high production rates, they cause the dies which are commonly made of H13 die steel, to suffer frequent failures. The major die failure mechanisms are erosion or washout, Heat checking, soldering and corrosion. Due to their geometrical complexity, die casting dies are very expensive (some dies cost over a million dollars), and thus a large number of parts have to be produced by a die, to justify this cost and leverage the advantages of the die casting process (high production rates, low manpower costs). A potential increase in the die service life, thus has a significant impact on the economics of the die casting operation.

There are many ways to extend die life: developing new wear resistant die materials, developing new surface treatments including coatings, improving heat treatment of existing H13 dies, using better lubricants that can protect the die material, or modifying the die geometry and process parameters to reduce the intensity of wear. Of these the use of coatings to improve the wear resistance of the die surface has shown a lot of promise. Consequently, use of coatings in the die casting industry and their wide use to decrease die wear can improve significantly the productivity of shop operations resulting in large savings in material and energy usage.

Past research at the Ohio State University has focused on investigating the wear mechanisms, development and evaluation of coatings for die casting dies by controlled

laboratory experiments. However, for the large scale adoption and use of coatings in the industry, the efficacy of coatings need to be proved amidst the numerous factors which arise in day-to-day production environment which no laboratory test or computer simulation can simulate. Therefore the testing must be done in-situ. The primary goal of this project was to evaluate selected candidate coatings in production environment and generate data on their long term use (approx. 100,000 shots). Such long term production data would provide the needed confirmation of the beneficial impact of surface coatings in increasing die lives. This project involved a comprehensive set of activities which included a literature and industrial survey on the use of coating, laboratory and production evaluation of the selected candidate coatings, coating optimization, and guidelines for the selection and use of coatings.

The project was sponsored by DOE (Office of Industrial Technologies) at the Ohio State University in association with the North American Die Casting Association (NADCA) Coating and Surface Treatments Task Group, and a consortium of die casting, coating, heat treatment and steel companies.

The results from this study show that the use of coatings can result in up to 10X increase in die life leading to savings of about \$25,000 on one campaign of 100,000 shots. This report summarizes both the state of die coating usage in industry and the extensive research results, both published and unpublished. It includes (a) background on wear mechanisms in die casting, (b) an overview of coating application techniques and general requirements of a good coating material, (c) a comprehensive review of

corrosion studies, erosion studies and thermal cycling tests conducted at Ohio State, (d) results from mechanical cycling tests conducted at Ohio State to determine fatigue data (based on the method of universal slopes), Young's modulus data for coating systems, and fracture data, (e) results from the production evaluation tests that have given 10X increase in die life and which clearly point towards the savings and increased productivity that can be achieved by the use of coatings, (f) summary of an industrial survey on the use of coatings in die casting, and (g) emerging directions in research and coating optimization.

Chapter 1: Introduction And Background On Die Wear In Die Casting

1.1 Introduction

The die casting process is a high volume production process which produces geometrically complex parts of nonferrous metals with excellent surface finish to net or near net shape. Production rate of 200 parts per hour and production batches of 300,000 parts are not uncommon. Currently, more than 50% of die castings manufactured are used in automotive applications. Non-ferrous metals such as aluminum are capable of producing the excellent mechanical performance often necessary for demanding vehicular applications. This allows the replacement of steel components by the lighter aluminum and magnesium components, providing overall weight savings for the vehicle. In terms of energy, even a pound reduction in the average weight of a car results in a really significant reduction in the gasoline usage. It is predicted, in a recent study, that an average of 155.2 lb. of aluminum die castings will be used in each automobile in the year 2010. This study assumes that 15.93 million automobiles would be produced in the US and Canada in the year 2010.

The U.S. die casting industry consists of about 400 production facilities which include a few plants at the OEMs and a large number of small manufacturers. Die casting is the second largest segment of the casting industry with annual production of eight billion dollars worth of castings per year.

1.1 Energy Consumption in Die Casting

Die casting is an energy intensive manufacturing process in terms of Electricity, Natural Gas, Propane and Fuel Oil. With the expected increase in the average usage of aluminum die castings per car, it becomes imperative to make the process more energy efficient. The following is an industry wide energy provided by the North American Die Casting Association(NADCA).

Energy Form	Quantity	Conversion($\times 10^6$)	Energy ($\times 10^{12}$ Btu)
Electricity	8.7×10^8 Kwh	0.0105 Btu/ Kwh	9.135
Natural Gas	2.2×10^7 ft ³	0.001 Btu/ ft ³	0.022
Propane	7.4×10^6 gal	0.150 Btu/ gal	1.11
Fuel Oil	3.9×10^5 gal	0.139 Btu/ gal	0.0521

$$\text{Total Energy Consumption} = 10.3191 \times 10^{12} \text{ Btu}$$

Table 1.1: Energy usage in die casting industry

From the table above, it is evident that even a small percentage of energy saving could lead to significant economic benefits by reducing soldering and washout resulting in reduced scrap rates, down times, washout rates, and increased die lives.

In addition to the above energy utilization, die casting of aluminum or magnesium also consumes energy indirectly. For example, production of aluminum itself is very energy intensive which starts with the reduction of aluminum from bauxite. A large fraction of

cost of aluminum (aluminum alloys range from \$0.70 to \$1.00 per pound or more depending on composition and processing) is for the energy consumed in processing. Also, other consumables such as lubricants, steel dies, etc. consume additional energy. Therefore, greater productivity and lower scrap rate in die casting will yield several multiples of savings in energy use.

1.3 *Critical costs in die casting*

The die casting process involves the use of permanent steel molds typically made of expensive H-13 die steel to produce castings continuously using the same die and machine. While such an arrangement provides for high production rates and efficiencies of labor, it also involves high startup costs. Dies are typically very expensive (often costing up to a million dollars) and machines capable of producing the high pressures (2000 psi is common) and velocities (60 m/s) are required. Consequently the die casting process becomes viable from a cost/part standpoint only when a very large number of castings are required (50,000 - 100,000) to be produced. Because of the high cost of dies, an extension of die life will consequently have a significant impact on the economics of the die casting operation. Cost of downtime for a die casting machine is extremely high. Therefore, reduction in down time can make the critical difference between loss and profit for the casting manufacturer.

1.4 Description Of The Physical System

Die castings are generally produced by using two steel die halves called the cover and ejector die halves. Each of the die halves usually contain a portion of the die cavity. The process sequence of a typical die casting process is described in figure 1.1. The die casting cycle consists of the following steps.

1. Lubrication: The open die halves are sprayed with water based lubricants and anti solder compounds.
2. Die Closing: The die halves are closed and locked by the die casting machine. The required clamping force during the process may be in the order of hundreds of tons.
3. Cavity Filling: The next step involves the injection of the molten metal into the die cavity under very high pressures. Rapid filling of the die cavity is desirable as it reduces the chance for premature solidification that could shut off the metal flow to portions of die cavity. Typical filling times are in the order of milliseconds with typical gate velocities of the order of 2000 in/sec.
4. Casting Solidification: The molten metal is then allowed to rapidly solidify in the die cavity. Heat management is critical in this step as proper location of cooling channels will help promote directional solidification of the casting and consequently reduce shrinkage porosity. Metal pressure intensification at the end of the injection cycle is sometimes used to feed shrinkage.
5. Parts ejection: After the casting has solidified, the die halves are opened and the part is ejected.

The above process sequence repeats with lubricant spray which causes the die surface temperature to drop drastically.

1.5 Die-casting interface activity

The plane of separation between two phases is known as an interface. During the different stages of the die casting operation the following activities take place at the die surface which cause formation of different interfaces. Considering ideal die casting conditions the formation of various interfaces is shown in Fig. 1.2 and discussed below.

Start up stage : The first interface formed on the die material (usually H-13 steel) is the oxide layer of the die cavity surface. It is known that the oxidized die surface can prolong the service life of the die by acting as a barrier and preventing the direct interaction of the molten metal with the die surface. Surface heat treatments like micropeening, nitriding etc.⁷ and hard coatings are also used to increase die life⁸.

Lubrication stage : During the lubrication stage, a steam layer is formed between die surface and lubricant droplets as the lubricant spray hits the hot die surface. This prevents the lubricant from reaching the cavity surface. However, as the spray continues the die temperature drops to a wetting temperature and a lubricant film is formed. Some die casters follow the lubricant spray with a drying cycle to reduce the gases due to lubricant carrier evaporation.

Cavity filling stage: During cavity filling stage the molten metal enters the die cavity at high velocities through the gates in an "atomized" form¹⁰. This molten metal further evaporates the lubricant leaving a layer of solute. It also washes out lubricant layer from the cavity surfaces near the gates or surfaces perpendicular to the jet-stream.

Casting solidification stage : During the solidification stage the melt rapidly dissipates heat and solidifies. Hot metal contact may cause thermo-chemical reactions at the cast metal-die surface interface. After solidification, the cast metal may separate from the die surface due to volume contraction.

Casting ejection stage: The casting now cools from the solidification temperature and shrinks from the die surfaces. Depending on the orientation of the surface, it either shrinks on-to or away-from the cavity surface. Ejection of this hot casting results in (a) soldering at hot spots, (b) drag marks at cores and sliding surfaces and (c) friction at surfaces with no lube separating the casting from the die surface.

1.6 Die Wear in Die Casting

Low cycle times in die casting dictate that molten metal be introduced into the die cavity at high flow velocities, and that the molten metal rapidly solidify (large thermal gradients in die cavity). Flow velocities of 40 m/s (132 ft/sec) and die temperature gradients as high as 1000 C/cm (4500 F/in) are typical. While these severe conditions are mandated to achieve these high production rates, they also limit the die materials that can be used and their respective production campaign. Various wear phenomenon are widely observed in H-13 die steel, the most commonly used die material due to this severe mechanical and thermal loading. Major wear mechanisms leading to premature die failure or causing large die downtimes are described below.

1.6.1 Erosion or Washout

Washout near the gates is a result of the high velocities with which the molten metal impinges parts of the die cavity, causing the steel to be eroded away with the melt.

Most die casting dies have complicated geometrical features such as cores, pins, ribs and corners which are especially prone to erosive wear. This reduces the ability of the die to maintain dimensional tolerances, and often requires rebuilding of regions of the die that have suffered extensive washout.

1.6.2 Heat Checking (*thermal cracking*)

Thermal cracking is caused by the thermal fatigue due to the alternate heating and cooling of the die surface during die casting. The large thermal gradients present cause the die surface to be in compression during heating, and the tension during cooling of the die. This results in thermal cracking which appears as cracks called heat checks on the die surface which reduce part surface finish, and ultimately lead to die failure.

1.6.3 Soldering and Corrosion

Soldering caused by the thermo-chemical interaction of the casting alloy and the die material during solidification. This results in parts of the molten metal sticking to the die surface (soldering) which produces defective castings, or part of the die surface being corroded. Frequent production stoppages to clean the soldered areas cause significant increases in die/machine downtime and production losses.

As the above discussions indicates that the thermo-mechanical-chemical interactions at the casting-die interface result in production losses. (1) corrosive wear causes defective castings, difficulty in ejection due to aluminum sticking to the components, (2) erosive wear causes the die component (and hence the casting) to go out of specification and (3) heat checking is a critical problem (in large dies especially) with respect to the

surface finish and ultimate failure of the die. The combination of these cause downtime in machine shops (a) to clean up the dies and components and (b) to replace inserts.

The objective of a long term research program at Ohio State is to extend die life so as to prevent the problems identified above. Several options present themselves:

1. Develop new wear resistant die materials
2. Develop new surface treatments and coatings
3. Improve the heat treatment of existing H-13 dies for higher toughness
4. Develop better lubricants that can protect the die material
5. Redesign of die to reduce thermomechanical loads
6. Process parameter modifications to reduce die wear severity

Of all the options above, it is apparent that some like the redesign of die, are extremely difficult to carry out such as are developing new cast or die materials. The best alternative easily remains the use of coatings and surface treatments. The main advantage of this option is that the die design, the process parameters, etc. can be retained as is. Coatings thickness are typically of the order of few microns and hence do not affect the tolerances desired on core pins (and hence on the cast components). At the same time, the benefits it offers are tremendous: life increases of over 10X, reduced clean up and consequent energy and monetary savings.

1.7 Selected Coating and Surface Treatment Techniques

In coating deposition processes, material is added to the surface, while in surface treatments the surface microstructure and / or chemistry is altered¹. PVD, CVD, PCVD, TRD, Thermal Spray, etc. are some examples of the former, and carburizing, nitriding, cold working, work hardening, etc. are examples of the latter. Figure 1.3 includes the broad classification of coating techniques and surface treatments.

The selection of a particular coating process depends on numerous requirements which include the size, shape and properties of the substrate, the purpose of the coating, the adhesion level needed and cost¹. Three major techniques have found a wide acceptance in die wear applications: Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD) and Thermo-Reactive Diffusion and Deposition (TRD).

PVD processes are those in which the process of transporting an atom or molecule to the surface being coated is accomplished by a physical process². These are line-of-sight processes and thus coating complex parts is difficult. However, the lower (100-500C) temperatures of application make them very versatile with respect to the types of substrates that can be used. They include such processes as vacuum evaporation, sputter deposition and cathodic arc deposition.

CVD processes are those that use chemical reactions to deposit the coatings onto the substrate. Typically, these are carried out at higher temperatures (150-2200C) thus reducing the number of substrates that can be coated by this process. However, the

nature of technique (non line-of-sight) makes it easy for even complex components to be coated besides producing coatings with good adhesion.

TRD is a process that is essentially metallurgical using a simple heat treatment furnace³. Various carbides and nitrides can be deposited by this process. The substrate temperatures vary between 540C (Low temperature TRD) to 1025C (High temperature TRD).

Further details of the various types of coating techniques, coating material, substrate conditioning, etc. are beyond the scope of this report, and can be obtained from Refs. 1,3 and 4 .

Table 1.2 shows some of the typical coatings that are used in die casting and their application techniques. This list is not intended to be comprehensive or complete in all respects but to serve as a basic guide only, and thus only the better known coatings in die casting and their application techniques were included.

Application Technique	Coatings
PVD	CrN, TiN, CrC, TiAlN, ZrN, VC, etc.
CVD	CrC, TiN, ZrN, VC, etc.
TRD	VC, CrC, CrN, etc.

Table 1.2: Some commonly used coatings for die casting dies and their application techniques

1.8 Coating selection guidelines

Given the variety of processes and coatings available, the question arises as to how the selection of a coating should be made. Bhushan⁵ listed the requirements of a good coating material as follows:

1. Adequate adhesion to the substrate for longer life
2. Good mechanical properties (hardness, ductility, fatigue strength, shear strength and ultimate tensile strength)
3. Corrosion and oxidation resistance
4. Shock resistance - thermal and impact
5. High thermal conductivity to dissipate heat from the interface quickly
6. Dimensional stability
7. Thermal properties compatible with the substrate to minimize interface stresses
8. Compatibility between the coating and substrate
9. Low surface wear

These requirements provide a good starting point for the selection of a coating material for any application, but it must be realized that it is meant to be a general guideline

only. No one coating will satisfy all those requirements and no one coating can be used in every application. Stratford et al.⁶ state that the coating must be able to withstand the environment and thus protect against any wear that this environment is responsible for. At the same time it is necessary that the coating be compatible with the substrate. Both criteria must be taken into account while selecting a coating. Tables 1.3 and 1.4 summarize the requirements for both the coating and substrate so that a compatible system can be designed.

	Property	Requirement
Mechanical Properties	Hardness	High
	Strength (comp. & tens.)	High
	Ductility	Moderate
	Toughness	Moderate
	Elastic Modulus	High
Physical Properties	Thermal Conductivity	High
	Coeff. Of Thermal Exp.	Low
	Melting Point *	High
	Coeff.. Of Friction **	Low
	Density (Porosity)	High
	Crystal Structure	Simple, High Symmetry
Chemical Properties	Chem. Compatibility ***	Good
	Composition	Close to Stoichiometric

* Relative to operating or developed service temperature

** Relative to opposing sliding surface (counterface)

*** Relative to counterface or environment

Table 1.3: Property Requirements for an Ideal Coating.⁶

Properties	Requirements
Coeffs. Of Thermal Expansion	Low, Matching
Thermal Conductivities	Matching *
Elastic Moduli	High, Matching
Chemical Compatibility	High interfacial bond strength and need to promote alloying **

* Thermal barrier coatings should possess low thermal conductivity

** A favorable (negative) free energy of coating / substrate interaction should be accompanied by favorable (slow) kinetics of interdiffusion for service in situations where temperatures are operative or are developed

Table 1.4: Property Requirements for Coating and Substrate for an Optimal Coating System.⁶

Chapter 2: Evaluation of Coatings

2.1 *Controlled / Laboratory Tests*

This chapter provides an overview of some of the more important laboratory studies on die wear and coating evaluation in controlled environment conducted recently to investigate the individual wear phenomenon.

2.1.1 *Corrosion Studies*

To investigate the dissolution and soldering mechanisms of both coated and uncoated H13 die steel, an experimental procedure was developed at Ohio State. This set up (referred to as dip-test set up) consists of a furnace with a temperature controller in which a crucible is placed. The crucible was chosen to accommodate at least four pins (later upgraded to six to eight pins) by the means of a fixture on the crucible. A structure mounted with a motor linked to the fixture on which the pins rest allows it to be rotated at desired speeds. Details of the test set up can be found in Appendix A (and also from Refs. 7 and 8).

Investigations done on uncoated H13 die steel showed the formation of several Al-Fe-Si intermetallic compounds which resulted from multi-element diffusion between the H13 and molten A390 melt. This aggressive attack was found to depend on the melt temperature and melt velocity⁸. The rate of dissolution of H13 in a rotating melt was found to be higher than in a static melt. Increase in temperature also contributed to

acceleration in corrosive attack by a factor of 2.5 when the temperature was increased from 610 to 750 C.

The effect of various important parameters on the corrosion and soldering tendencies were examined such as: the effect of heat treatment, effect of surface coating and casting alloys⁷. The heat treated pins (with HRC: 42, 49 and 52) showed slightly improved performance over pins without heat treatment. There was no significant difference in corrosion resistance between heat treated pins of different hardness values. The coatings that were tested included TiN (PVD), VC (TRD), TiN (CVD), B₄C (CVD) and experimental coatings from NASA. A heat treated H13 pin was used as a control pin. Among all the coatings VC demonstrated the best corrosion and soldering resistance. Casting alloys that were tested included commercially available pure Al, Al413 and Al390. It was found that Al390 showed the most corrosive action and pure Al provided the least corrosive attack. More detailed discussion on the causes and cures of the corrosion and soldering mechanisms in aluminum die casting can be found in Refs. 7 and 9.

2.1.2 Erosion Studies

To adequately evaluate die erosive wear, an accelerated test was developed at Ohio State which included a multi-pin test die and aggressive process conditions. The die was basically a flat plate with six core pins, four facing the gates and the remaining two placed behind these four. The die was run on the Buhler 250SC machine. The front row of four pins were used for the relative evaluation of coatings. The wear of the core pin

surfaces was chosen to represent the accelerated wear of die surface exposed to high flow velocities. In addition, a pyramidal test pin design (with sharp edges) was selected for increased erosive wear rate and to aid the measurement process (straight edges are easy to measure compared to rounded surfaces).

Numerous studies were undertaken at Ohio State with this test die. The effect of process parameters on material removal was examined in detail including the effect of metal velocity, angle of metal impact and metal temperature^{10,11}. The weight loss (measure of wear) was found to be directly proportional to inlet gate velocity. Pin surface inclination of about 72 degrees to the melt stream was found to show highest erosive wear. Erosive effects were found to decrease with increase in melt temperature, this may sound contrary to the results reported in corrosive dip tests (in the previous section). However, the dip tests do not take into account the fact that at higher temperatures the solid fraction in the melt decreases. This solid fraction is primarily due to premature solidification in the shot sleeve, biscuit and the runner system. Consequently, the impact of solid particles on pins that is present at lower temperatures is lessened.

Numerical model based on FLOW-3D software was developed to simulate the transient, inertia dominated complex filling in high pressure die casting. This model has the capability of handling free surface profiles, transient flow, jetting and splashing which occur during the isothermal filling of die cavities¹². Numerical results were used

to derive models of erosive wear and to investigate the fundamental variables of erosive wear.

Specific experiments were also conducted to study the erosive wear of coated and surface treated die surfaces. Erosive performance of 16 coatings and surface treatments was evaluated using the multi-pin accelerated test. These included TiN, VC, W, Pt, Mo, MetalLife and NASA coatings. The PSII coatings (Mo, W, Pt) were included in this study since they were reported to have performed very well in thermal fatigue tests at Case Western Reserve University. However, their resistance to erosive wear was not as high as ceramic coatings with only the W coating performing better than the uncoated H13 pin. Among the coated pins, VC performed the best with the least amount of wear. Other coatings such as TiN performed well too, with definite improvement shown over uncoated pins. Test set up and other details of results are available in Appendix B.

Based on the above results a CrC coating (applied by the pack cementation process) was developed for die casting. This coating had good erosive wear resistance and adequate resistance to heat checking due to good match of coefficient of thermal expansion with die steel substrate. Yu⁸ reported a decrease in erosive wear by a factor of 4 in accelerated erosion tests (up to 650 shots) with this CrC coating. More details can be obtained from Refs. 10-14.

2.1.3 Mechanical Cycling Tests

There are several critical issues in the analysis of any coating system (the coating / substrate pair). These include: What are the coating and substrate properties? What does the coating process do to the substrate? What is the best substrate to be used? Rosbrook and Shivpuri¹⁵ had shown that the method of universal slopes could be used to successfully predict the crack initiation of H13 tool steel subjected to a thermal cycle representative of a die casting operation. However, the method of universal slopes is based on the material properties of the coated specimen being known. Since, coating systems are made of two materials, this data is not readily available and needs to be experimentally determined.

An investigation was conducted at Ohio State¹⁶ whose main objectives were to:

1. Develop an experimental procedure that will characterize coating systems in terms of their performance under the mechanical cyclic loading seen in high pressure die casting
2. Use this procedure to develop fatigue data based on the method of universal slopes for various coating and substrate combinations
3. Experimentally develop Young's modulus data for different coatings and coating systems (needed for thermal fatigue calculations)

Following the logic of Rosbrook and Shivpuri, this investigation concentrated on the forces caused by the cavity pressure, and treated surface stresses due to locking forces as negligible. FEM simulations showed that the die under these conditions will be subjected primarily to bending. Thus, it was decided to run cyclic bend tests. A four

point bend test was chosen since it was found that the portion of the specimen between the two inner loading points is in pure bending¹⁷. The geometry of the fixture and the exact positioning of the bending points is shown in Figure 2.1. These were based on recommendations obtained from ASTM standards on four point tests used for advanced ceramics¹⁸. The test fixture was attached to a MTS fatigue machine for loading and cycling.

The substrate used in the testing was H13 die steel, since its the most widely used die steel. The steel was heat treated to two different hardness levels (Hard= 48-52 HRC and Soft= 42-46 HRC). Three different coatings [VC (TRD), CrN (PVD) and CrC (PVD)] and one surface treatment [nitriding] were selected. Load vs. Strain data was obtained experimentally for each of the eight coating systems (4 coatings and 2 substrates) tested. From these, Young's Modulus estimates were made for each of these coating systems and are shown in Table 2.1.

Coating/Surface Tr.	Coating Company	Hard Substrate	Soft Substrate	% Difference
Nitrided	Advanced Heat Treat	38.4x10 ⁶ psi	39.1x10 ⁶ psi	1.82%
VC	Arvin TD	35.4x10 ⁶ psi	38.6x10 ⁶ psi	9.04%
CrN	Multi-Arc Inc.	35.1x10 ⁶ psi	36.3x10 ⁶ psi	3.42%
CrC	Balzers	37.7x10 ⁶ psi	39.1x10 ⁶ psi	3.71%
% Difference btwn Max and Min		9.40%	7.71%	-----

Table 2.1: Young's Modulus Measurements for Coating Systems

2.1.3.1 Fatigue Testing data

The testing scheme has been summarized in Table 2.2:

Coating System	Fatigue Data Results
Hard Substrate	
Nitrided	Low and high cycle data obtained System never reaches plastic region; always elastic Strain history was 'elastic'
VC	Low and high cycle data obtained System never reaches plastic region; always elastic Strain history was 'elastic'
CrN	Low and high cycle data obtained System never reaches plastic region; always elastic Strain history was 'elastic'
CrC	Low cycle data obtained (plastic) Used completely reversed strain history Still need high cycle data
Soft Substrate	
Nitrided	Obtained one point in plastic region Obtained one point in elastic region Strain history was 'initially plastic, elastic'
VC	Fractured at loading points Used completely reversed strain history
CrN	Fractured at loading points Used completely reversed strain history
CrC	Fractured at loading points Used completely reversed strain history

Table 2.2: Summary of Fatigue Testing using Four Point Bend Test Fixture

There were three kinds of strains that the specimen experienced:

1. Elastic cycle: Some of the systems tested did not reach plastic strains in the low cycle regime. This allowed completely elastic cycling of these specimens. Figure 2.2 shows this kind of strain history. The strain was unloaded after every cycle.
2. Initial Plastic, Elastic Cycle: The load, in some cases, caused the specimen to yield on the first cycle. The plastic strain does not unload and after strain hardening on the first cycle the specimen experiences a purely elastic cyclic strain range. This strain history is shown in Figure 2.3.
3. Completely Reversed Cycle: Some of the specimens were flipped over after every cycle. This was done to completely reverse the strain in the specimen. In order to ensure that the same strain was attained after every loading, the machine was advanced a certain displacement. Figure 2.4 depicts this strain history but is strictly not accurate since the specimen would stay at a particular strain while being manually flipped over.

The fatigue experimentation results have been plotted in Figs. 2.5, 2.6, and 2.7 as a function of applied strains. The results are compared with the universal slopes prediction for H13. This model of H13 was based on experimental data. However, to make accurate comparisons, H13 at the different hardnesses, used in these experiments, needs to be tested.

In Figure 2.5, it may be noted that all the three systems [VC (TRD), CrN (PVD), Nitriding] have approximately the same slope and behavior, just at different strain

levels. These systems perform worse than H13 at low cycles. However, at about 10,000 cycles their performance becomes comparable to H13, and this model predicts better performance in high cycle regimes. The region that applies to die casting is approximately between 100 and 100,000 cycles. At the higher end of this region, the coated systems performs is comparable to H13. Figure 2.6 shows the result of the fatigue tests on the Nitrided pin with the soft substrate. Two points were obtained for this system. One that fractured at the other cycle, and one at 1925 cycles. The former was in the plastic regime before it fractured and the latter was in the elastic. Thus, an accurate estimate of this system is not possible but the plot gives an approximate idea of where this system would be on the fatigue plot. Figure 2.7 shows the CrC (PVD) pin on a high hardness substrate which experienced a completely reversed strain cycle. This line has a smaller slope when compared to the universal sloped prediction for the plastic strain for H13. This means that the total strain curve for this system will most likely be below that for H13 in the low cycle regime. However, for 1000 cycles and higher this system will probably have better performance than H13 alone. This data needs to be developed.

2.1.3.2 Fracture Analysis

The fractured specimens were all examined macroscopically. Further examination might be necessary to firmly make conclusions concerning fracture. Table 2.4 shows the type of fracture observed in the tested specimen.

Coating / Heat Treatment	Type of Fracture Observed
Nitrided (hard substrate)	Brittle fracture
Nitrided (soft substrate)	Brittle fracture
VC (hard substrate)	Brittle fracture
CrN (hard substrate)	Brittle fracture
CrC (hard substrate)	Both brittle and ductile, seen at different points

Table 2.3: Type of fracture observed in selected coating system specimens

Figures 2.8 (a) and (b) show an example of a fractured specimen. For all the cases (except the VC specimen, where fracture initiation could not be located), fracture initiated in the substrate below the coating. A possible interpretation for this is that the coating causes a substantial shift in the neutral axis of the substrate due to the high Young's modulus of the coating. This would make the strains quite a bit higher at the bottom of the specimen when compared with the top, and thus making the initiation of fracture more likely at the bottom. The hardness profile data was taken of the fractured pieces through their thickness. This was done to find out the depth of the coating affected zone for each system. However, this data did not prove very useful.

2.1.4 Thermal Cycling / Cyclic Oxidation Tests

Cyclic oxidation tests were conducted at Ohio State with selected coatings. These tests showed that Coefficient of Thermal Expansion (CTE) of coatings played an important

role in the thermal cycling characteristic of coatings. A match in the CTEs of the coating and the substrate was found to be important for thermal fatigue resistance. The Table 2.5 shown below shows the CTEs of some of the coatings tried at Ohio State⁸.

Material	CTE(10^{-6} C^{-1}) at 650 C
H13 *	7.3
Cr ₂₃ C ₆ (CrC)	7.6
VC	4.4

* Commercial H13 die steel with HRC 46

Table 2.4: CTE values of selected materials⁸ (CrC and VC were applied by pack cementation and salt bath techniques respectively)

The cyclic oxidation test involved placing coupons into a quartz holder that was separated with an alumina-base refractory paper. The samples were lowered into the hot zone of the furnace for 30 minutes then pulled out and air-cooled for 30 minutes to finish one oxidation cycle. A typical oxidation campaign was 300 cycles. The coatings tested were VC (salt bath) and CrC (pack cementation) along with plain H13. It was seen that CrC performed the best with the least amount of weight pick up due to oxidation. The cyclic oxidation / thermal cycling tests indicated that the CrC coating possessed excellent oxidation resistance and thermal cracking resistance at temperatures in the range of 550 -700 C. It was seen that the coatings that performed best with respect to thermal cycling were the coatings which had a closer match of the CTE with the substrate.

2.2 : Candidate Coating Selection

An important objective of this task was to identify clearly the candidate coatings that were to be used in the testing phase of the program. To enable the selection, the short listing was based on two main criteria: first, a comprehensive industry survey of coating usage to identify the most popular and effective coatings being used in the die casting industry and second, the results of the coating evaluation carried out previously at Ohio State and elsewhere. The most promising of all the coatings that figured in the two sets of data were to be the candidates for production testing.

Previous tests conducted at The Ohio State University were either near-production type (erosion tests) or simulated laboratory type (corrosion tests). These tests helped screen potential coatings, which had the capability to enhance wear resistance of H-13 dies. However for the large scale adoption and use of coatings by the die casting industry, the efficacy of coatings need to be proved amidst the numerous factors which arise in day-to-day production environment. Some of these factors are:

1. Robustness of coating to varying process conditions during production
2. Ease of coating application and cost
3. Repairability of coatings and coated dies
4. Susceptibility of coating to nicking, scoring, etc.
5. Coating-lubricant interaction

There is no laboratory test or computer software than can simulate these factors before trying out coatings in production. Therefore the testing must be done *in situ*.

2.3 Industrial Survey

As part of the effort to compile detailed data on the use of coatings in the die casting industry, a survey was sent to 53 die casters and 4 coating companies. The questionnaire was designed to collect information on specific case histories. Appendix C contains the details on the initial surveys including the format, questions asked and sample responses. Appendix D has the updated questionnaire and a sample response from a die caster. The following table 2.5 summarizes the results of the survey.

Detail	# of companies
Number of survey responses/sent :	
Die Casting companies	31 out of 53
Coating Companies	3 out of 4
Major component manufactured: Auto parts	20
Others	11
Substrate Material: H13 (premium grade)	11
H 13	20
Production Volume: Up to 100,000	14
> 100,000	6
Coatings used: Chromium Carbide	12
Chromium Nitride	10
Titanium Nitride	5
Titanium Aluminum Nitride	4
Vanadium Carbide	1
Others	1

Note: One company reported significant improvement in wear resistance by the use of double coatings

Table 2.5: Summary of industry survey on the use of coatings in die casting

Based on the performance data from industry and the laboratory testing, the coatings identified to be the most promising with respect to the wear resistance in die casting die application were the following (table 2.6):

Coating Material	Coating Process
Chromium Carbide (CrC)	Physical Vapor Deposition
Chromium Nitride (CrN)	Physical Vapor Deposition
Vanadium Carbide (VC)	Thermo-Reactive Deposition
Titanium Nitride (TiN)	Physical Vapor Deposition

Table 2.6: Coatings selected for production testing and their application technique

Chapter 3: Research Objective and Plan

3.1 Objective

The laboratory evaluations carried out at Ohio State and elsewhere pointed to the superior performance of coated samples compared to uncoated ones. These tests also indicated that the coating types performing best in different tests were different. No single coating performed best in all the tests (corrosion, erosion and thermal fatigue). It was also understood that while these accelerated tests were very good indicators of the performance of a coating, they did not consider simultaneously the effects of corrosion, erosion and thermal cycling as is often the case in production. In addition, the production environment is sufficiently different from the laboratory environment to warrant production validation of test results. The contract objective of the project was:

"To use one or two commercially available coating technologies to apply coatings to die casting die components, inserts or cores, and to evaluate the effectiveness of selected coatings in improving casting quality."

The research team decided to evaluate pre-screened coatings in production environment and generate data on their long term use (approx. 100,000 shots). The testing strategy was to obtain the average die life data by randomized experimental design procedure. An industrial monitoring team was established with the help of the NADCA Die Coatings & Surface Treatment Task Group (Mr. Peter Ried, Doehler-Jarvis, Chairman). The industrial participation was in terms of providing beta test sites, sample coatings, heat treatment services and die steel test specimens.

3.2 Operational Plan

The first step in the planning was to identify the main activities that need to be performed. The *Activity Chart* shown in figure 3.1, summarizes the testing and evaluation procedure. Once the potential die caster was identified, the test die was selected based on the criteria described earlier. The core pins/inserts are then obtained from the die casting company. Each of the core pins were assigned an identification code based on the company, testing batch and coating to be applied. If the pin needed to be heat treated, it was sent to the heat treating company and was heat treated according to NADCA heat treatment guidelines. The coded pins were then sent to the coating companies for coating application. The average turn around time for coating application was 4 weeks. A pre-testing surface evaluation was performed and the surface roughness values were obtained. These details were documented and a *Campaign Sheet* for each testing campaign was generated. The coated pins were then put in production and constantly monitored. On failure (as defined by shop floor personnel) the pins were removed from the die and the post-testing evaluation was performed. Figure 3.2 shows the *Subtask Chart*, which identifies the various participants and the individual tasks.

3.3 Project Schedule

Discussions were held with different die casting companies to identify potential beta sites. Table 3.1 and 3.2 summarize the efforts taken and the project time utilization. Discussions were held with die casters: General Die Casters, Pace industries, Inc., Doehler-Jarvis Technologies, and Ganton Technologies. Meetings were also held with

coating companies and agreements were made with Balzers Tool Coating, Multi-Arc, Inc., and Arvin TD for application and evaluation of coatings. Based on the turn around time, production schedules etc., the coating schedules were drawn up. Appendix E gives a list of all the meetings held in connection with this project.

3.4 Research Participation

The industrial team consisted of :

Organizations

1. NADCA Die Coating and Surface Treatment Task Group

Die Casting Companies

1. Pace Industries, Inc; Monroe City, MO
2. Doehler-Jarvis Technologies; Pottstown, NJ
3. Ganton Technologies, Racine; WI
4. General Die Casters, Cleveland; OH

Coating Companies

1. Multi-Arc Inc., Rockaway; NJ
2. Balzers Tool Coatings, North Townawanda; NY
3. Arvin TD, Columbus; IN

Heat Treating Company

1. FPM Heat Treat, Elk Grove; IL

COMPANY	ACTION TAKEN	FINAL RESULT
General Die Casters	Plant visit, discussion, part selection	OEM did not give permission to run the test
Ganton Technologies	Plant visit, discussion, part selection, Design of experiments	No test run
Pace Industries	Plant visit, discussion, part selection, Design of experiments, casting trials, failure analysis	Testing completed
Doehler-Jarvis, Pottstown	Plant visit, discussion, part selection, Design of experiments	Testing could only be scheduled for 1997

Table 3.1: Project Activities

Company	1995				1996			
	Qtr1	Qtr2	Qtr3	Qtr4	Qtr5	Qtr6	Qtr7	Qtr8
General Die Casters								
Ganton Technologies								
Pace Industries								
Doehler-Jarvis								

Table 3.2: Project time utilization

Recently, more die casters have shown intent in participating in future extension of this testing program (Anderson Die Casting, Ganton Technologies, Newton-Haven Die Casting Company and Kennedy Die Casting).

3.5 Die Selection

The die/part selection was based on the criteria defined by the industrial monitoring team:

1. Die should have multiple cavities
2. Die must have symmetry
3. Die must be well known by the die caster (process established)
4. Local die process conditions known and documented near test feature
5. Set-up frequency should be known
6. Downtime causes must be documented in rank order of frequency
7. Test core pin or insert geometry should be well known.

It was decided that the focus would be on die pins/cores as the wear locations. The selected core pins were coded and sent for heat treatment and coating application. The coatings were applied as per the process specifications of the coating supplier. Candidate coatings were applied using methods as described in Table 2.6.

Chapter 4: Production Evaluation Test Program

4.1 Introduction

As a result of the initial discussions held with Pace Industries, Inc., Monroe (MO), it was agreed that a die which was then in operation would be made available as a beta test site for the production evaluation. This was followed by a data collection on the die, process parameters and production rate.

4.2 Test Die and Process Parameters

The die which was selected to run the test was a two cavity *Body Valve* part die. A multi-cavity die was chosen for two main reasons, firstly it would provide multiple sites for comparative evaluation and secondly it would facilitate easy assembly and disassembly for measurement. The process parameters for the die were:

Part Name	: Body Valve
Number of Cavities	: 2
Machine used	: 600 ton
Cycle time	: 45 seconds
Casting alloy	: A380
Metal temperature	: 1280 F

The core pins were H-13 steel, heat treated to 46-47 HRC. A sketch of the die is shown in Figure 4.1. Prior to the use of the coatings, the cores had to be pulled out every 4

hours for cleaning the soldering material. This cleaning on an average took 30 minutes, i.e. 1 hour per an 8 hour shift. The life of the uncoated core pin was about 15,000 shots, after which the dies were disassembled and the cores replaced.

4.3 Design of Experiments

Since this multi-cavity die provided simultaneous comparative evaluation of all selected coatings, a scientific approach was devised to generate meaningful data. It was decided that a Latin Square Design will be used. This design helps block the process variabilities such as difference in batches of pins and flow variability at different die locations. This design is shown in Table 4.1. It should be noted that the design is a square arrangement. The Latin Square design eliminates two nuisance sources of variability; that is, it systematically allows blocking two directions. Thus the rows and columns actually represent two restrictions on randomization.

Location/ Batch	Location #1 (Insert #1)	Location #2 (Insert #1)	Location #3 (Insert #2)	Location #4 (Insert #2)
1.	CrC	VC	TiN	CrN
2.	CrN	TiN	CrC	VC
3.	TiN	CrN	VC	CrC
4.	VC	CrC	CrN	TiN

Note: The insert and location numbering is indicated in the master layout

Table 4.1: Test matrix for the production test

The *Campaign Sheet* (fig. 4.2) with the insert numbers and the pin numbers was used to track the pin history. The failure of a pin was recorded on a *Failure Sheet* for every batch of pins.

4.4 Testing

The coated pins were put into production according to the test matrix. The core pins were periodically monitored by the operating personnel at the die casting company and the first *in-situ* evaluation was done at 33,897 shots (fig. 4.3). A small amount of soldering was observed on the pins particularly in the narrow sections of the cavity. Figure 4.4 shows a sample core pin.

Subsequently, the first failure was reported at 78,505 shots. The failed core pin was Chromium Carbide (CrC) coated. However, it was also reported that the failure was partly due to mechanical damage. Figure 4.5 shows the failed CrC coated pin. This pin was replaced by a new CrC coated pin and the testing continued. No further failure was reported until the end of the production run at 127,500 shots (fig. 4.8). That means that no cleaning was required for the entire campaign of 127,500 shots. Figures 4.6 and 4.7 show the Chromium Nitride (CrN) and Titanium Nitride (TiN) core pins at the end of the production run. Unfortunately, the Vanadium Carbide (VC) coated pin was never returned for failure analysis. The next batch of the coated core pins are due to go into production at the start of the next production cycle with this die.

4.5 Failure Analysis

Visual examination disclosed that all the three core pins sent back for evaluation (127,500 shots) had acquired a black oxide layer on the head and the shank of the pin. Some amount of soldering was also observed at the evaluation area (part of the pin exposed to the melt).

The pins were cleaned in 10% Sodium Hydroxide solution for a duration of 24 hours to remove all the aluminum sticking to the core pins. A surface analysis was performed on the core pins along the length of the shank. Three readings were taken for each pin by indexing it through 120 degrees. The average Ra, roughness value of the three observations, was taken as the measure of *pin roughness after testing*. Table 4.2 compares the Ra values before and after the testing. Note the significant increase in roughness values for the TiN pin indicating failure of coating possibly due to washout. The surface analysis plots at zero degree indexing, for the CrN, CrN and TiN pins are shown in figures 4.9, 4.10 and 4.11.

Core Pin	Ra Value (Evaluation Length: 7 mm)	
	Before Testing	After Testing
Chromium Carbide	0.3521 μm	0.7112 μm
Chromium Nitride	0.4572 μm	1.0000 μm
Titanium Nitride	0.2556 μm	6.0000 μm

Table 4.2: Roughness values of pins before and after testing

An important observation was: the presence of pits in areas where the coating was still remaining, and the propagated grooves in areas where coating was completely worn out. This is an evidence of *pitting corrosion*, in which the pits initiate on the coated surface and slowly grow to become wear locations. To further analyze this phenomenon and study the mechanism a series of accelerated laboratory tests were performed and similar mechanism of coating failure was found.

4.6 Future Work

Ohio State has already started research on examining pitting mechanism of coating failure. Some exciting directions have emerged from this, with failed pins showing localized pitting. Pitting corrosion (or pitting) is a method of failure unique to substrates that have a passivating layer. Extensive testing is being conducted to determine two main effects: first, the growth of pits (pit density) with time and temperature and secondly determination / explanation of pit initiation. Criteria that are being investigated include solubility and galvanic action. The results from the test matrix will be used to improve and engineer the coating that will be able to provide resistance far superior to coatings currently available. The ultimate aim of this program is to improve die life by at least a factor of 10X for most die casting applications.

Chapter 5: Summary of Project Benefits

5.1 Research Summary

The primary goal of this project was to evaluate selected candidate coatings in production environment and generate data on their long term use (approx. 100,000 shots). Such long term production data would provide the needed confirmation of the beneficial impact of surface coatings in increasing die lives. This project involved a comprehensive set of activities which included a literature and industrial survey on the use of coating, laboratory and production evaluation of the selected candidate coatings, coating optimization, and guidelines for the selection and use of coatings.

5.2 Guidelines and Recommendations on the use of coatings

Table 5:1 gives a summary of performance of various coatings on a relative scale. The table was developed using the data obtained from the laboratory tests as well as the reported performance of coatings (both published and unpublished work) in the production scenario.

	CrN (PVD)	VC (TRD)	CrC (CVD)	TiN (PVD)	Metal- Life (SuTr)	W (Ion Impl)	Pt (Ion Impl)	Mo (Ion Impl)	H-13
Corrosion	H	H	H	M	L	NA	NA	NA	L
Erosion	H	H	H	H	M	M	L	L	L
Heat Checking	M	NC	M	M	M	H	H	H	L

Scale used: H- High Resistance, M- Medium Resistance, L- Low Resistance, NC- Not Conclusive, NA- Not Available

Table 5.1: Relative performance of coatings in die casting resistance applications.

The data from the first batch of production testing is certainly promising and has certainly thrown up some vital issues with regard to failure mechanisms of coatings. Apart from generating useful production data, it also brought out the many difficulties that have to be faced in running a full scale production evaluation. The subsequent tests, will help better understand the variables in production that effect coating life. The significance of this preliminary effort is the fact that the cost of the coatings was less than 10% of the savings made by the die caster, which makes coatings an economically viable solution to die wear problems.

5.3 Cost Savings

The use of coated pins completely eliminated the need for periodic cleaning of the cores and most of them led to an increase in die life by an order of magnitude of more than 10X (127,500 shots) in this particular case. This led to significant savings in terms of machine downtime and labor, in the order of \$24,642 for one die per 100,000 shots. A savings summary for the Body Valve die is shown below in Table 5.2.

S.No.	Detail	Before use of coating	After use of coating
1.	Cleaning schedule	1 hr./shift (30 min. after every 4 hr.)	Completely eliminated
2.	Core pin life	15,000 shots	1 pin: 78,505 shots 3 pins: >100,000 shots
3.	Cost savings in terms of labor and machine downtime for 100,000 shots	----	\$ 24,642

Table 5.2: Savings summary table [courtesy: Pace Industries, Inc.]

5.4 Budget / Match

The project budget and the industry match are summarized in table 5.3.

	DOE	Industry Participation	OSU
OSU **	41		43
Die Casting Companies		24	
Coating Companies		22	
Heat Treaters		7	
Steel Suppliers		1	
TOTAL	41	54	43

* One fellowship for 2 years was awarded as a match by the NSF Engineering Research Center for Net Shape Manufacturing at the Ohio State University

** Students supported by this project also received tuition and fee match from the Ohio State University

Table 5.3: Project Budget / Match summary

Acknowledgments

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1. Die Closing 2. Cavity Filling 3. Solidification 4. Part Ejection

Figure 1.1 Operating sequence for a cold chamber die casting process
(Lewis, Chou, Chen, Han, & Kim, 1987).

1. Initial/After part ejection

2. Lubrication

3. Injection or Cavity Filling

4. Casting Solidification

Figure 1.2: Schematic diagram of interface activity under "ideal" die casting conditions

Figure 1.3: Broad Classification of Coatings

Figure 2.1: Schematic of Loading and Fixture for Fatigue Experiments on Coating/Substrate Test Specimens

Figure 2.2: Elastic Cycle

Figure 2.3: Initial Plastic, Elastic Cycle

Figure 2.4: Completely Reversed Cycle

Figure 2.5: Fatigue Results of Systems Tested with Elastic Strain History

Figure 2.6: Fatigue Results of Systems Tested with Initial Plastic, Elastic Strain History

Figure 2.7: Fatigue Results of Systems Tested with Completely Reversed Strain History

Figure 2.8(a) : Side view of fractured specimen of VC on hard substrate

Figure 2.8(b): Fracture surface of VC on hard substrate

Figure 3.1: Testing and evaluation procedure flowchart

2000

Figure 4.1: Sketch of test die - Pace Industries, Inc.

CAMPAIGN SHEET
(Ejector - Half)

CAMPAIGN I

(Started on 02/23/96)

Figure 4.2: Campaign Sheet - Pace Industries, Inc.

Figure 4.3: First in-situ evaluation at 33,879 shots

Figure 4.4: Sample core pin - Pace Industries, Inc.

Figure 4.5: CrC coated core pin at 78,505 shots

Figure 4.6: CrN coated core pin at 127,500 shots

Figure 4.7: TiN coated core pin at 127,500 shots

Figure 4.8: Campaign I - Results

profileView by MetreX

File Name: Untitled CrC - 0
 Filter Type: GAUSSIAN - PC
 Filter Cutoff: 0.030 in
 Tilt Correction: Straight Line (Entire Trace)
 Trace Speed: 0.010 in/sec
 Evaluation Length: 0.315 in

Computed Parameters:

$P_t = 866 \mu\text{in}$	$R_a = 28 \mu\text{in}$
$R_q = 38 \mu\text{in}$	$R_p = 96 \mu\text{in}$
$R_v = 148 \mu\text{in}$	$R_{\text{max}} = 244 \mu\text{in}$
$A_r = 0.001517 \text{ in}$	$H = 99 \mu\text{in}$
$R_{sk} = -0.898$	$R_{ku} = 4.303$
$R_z = 227 \mu\text{in}$	$R = 34 \mu\text{in}$
$R_k = 77 \mu\text{in}$	$R_{pk} = 28 \mu\text{in}$
$R_{vk} = 77 \mu\text{in}$	$M_{r1} = 8 \%$
$M_{r2} = 84 \%$	$t_p(39 \mu\text{in}) = 4 \%$
$t_p(79 \mu\text{in}) = 33 \%$	$t_p(118 \mu\text{in}) = 78 \%$
$t_p(157 \mu\text{in}) = 93 \%$	$t_p(197 \mu\text{in}) = 98 \%$
$t_p(236 \mu\text{in}) = 100 \%$	$t_p(276 \mu\text{in}) = 100 \%$
$t_p(315 \mu\text{in}) = 100 \%$	$t_p(354 \mu\text{in}) = 100 \%$
$t_p(394 \mu\text{in}) = 100 \%$	$W_a = 143 \mu\text{in}$
$W_p = 382 \mu\text{in}$	$W_v = 264 \mu\text{in}$
$W_t = 646 \mu\text{in}$

Figure 4.9: Surface Analysis of CrC coated pin at zero degrees

profileView by MetreX

Tilt Correction: Straight Line (Begin/End) Comments: CrN-0

Trace Speed: 0.255 mm/sec

Evaluation Length: 7.000 mm

Computed Parameters:

$P_t = 51.7 \mu\text{m}$	$R_a = 1.6 \mu\text{m}$
$R_q = 3.4 \mu\text{m}$	$R_p = 5.0 \mu\text{m}$
$R_v = 24.0 \mu\text{m}$	$R_{\text{max}} = 29.0 \mu\text{m}$
$A_r = 0.115 \text{ mm}$	$H = 4.6 \mu\text{m}$
$R_{sk} = -4.121$	$R_{ku} = 24.670$
$R_z = 12.6 \mu\text{m}$	$R = 2.0 \mu\text{m}$
$R_k = 2.6 \mu\text{m}$	$R_{pk} = 2.5 \mu\text{m}$
$R_{vk} = 13.9 \mu\text{m}$	$M_{r1} = 17 \%$
$M_{r2} = 88 \%$	$P_c(-2.5 \mu\text{m}, 2.5 \mu\text{m}) = 0.43 \text{ 1/mm}$
$HSC(2.5 \mu\text{m}) = 2.14 \text{ 1/mm}$	$t_p(1.0 \mu\text{m}) = 2 \%$
$t_p(2.0 \mu\text{m}) = 6 \%$	$t_p(3.0 \mu\text{m}) = 12 \%$
$t_p(4.0 \mu\text{m}) = 26 \%$	$t_p(5.0 \mu\text{m}) = 58 \%$
$t_p(6.0 \mu\text{m}) = 87 \%$	$t_p(7.0 \mu\text{m}) = 92 \%$
$t_p(8.0 \mu\text{m}) = 94 \%$	$t_p(9.0 \mu\text{m}) = 95 \%$
$t_p(10.0 \mu\text{m}) = 96 \%$	$W_a = 4.6 \mu\text{m}$
$W_p = 10.5 \mu\text{m}$	$W_v = 8.4 \mu\text{m}$
$W_t = 19.0 \mu\text{m}$

Figure 4.10: Surface Analysis of CrN coated pin at zero degrees

profileView by MetreX

File Name: Untitled (TiN) - 0
Tilt Correction: Straight Line (Begin/End)
Trace Speed 0.255 mm/sec
Evaluation Length: 7.000 mm

Computed Parameters:

$P_t = 71.7 \mu\text{m}$	$R_a = 2.4 \mu\text{m}$
$R_q = 3.0 \mu\text{m}$	$R_p = 11.8 \mu\text{m}$
$R_v = 7.4 \mu\text{m}$	$R_{\text{max}} = 19.1 \mu\text{m}$
$A_r = 0.205 \text{ mm}$	$H = 8.0 \mu\text{m}$
$R_{sk} = 0.321$	$R_{ku} = 3.911$
$R_z = 12.6 \mu\text{m}$	$R = 5.2 \mu\text{m}$
$R_k = 8.1 \mu\text{m}$	$R_{pk} = 4.8 \mu\text{m}$
$R_{vk} = 3.4 \mu\text{m}$	$M_{r1} = 6 \%$
$M_{r2} = 91 \%$	$P_c(-2.5 \mu\text{m}, 2.5 \mu\text{m}) = 1.43 \text{ 1/mm}$
$HSC(2.5 \mu\text{m}) = 3.86 \text{ 1/mm}$	$t_p(1.0 \mu\text{m}) = 0 \%$
$t_p(2.0 \mu\text{m}) = 1 \%$	$t_p(3.0 \mu\text{m}) = 1 \%$
$t_p(4.0 \mu\text{m}) = 1 \%$	$t_p(5.0 \mu\text{m}) = 2 \%$
$t_p(6.0 \mu\text{m}) = 2 \%$	$t_p(7.0 \mu\text{m}) = 4 \%$
$t_p(8.0 \mu\text{m}) = 7 \%$	$t_p(9.0 \mu\text{m}) = 17 \%$
$t_p(10.0 \mu\text{m}) = 29 \%$	$W_a = 0.7 \mu\text{m}$
$W_p = 2.4 \mu\text{m}$	$W_v = 1.1 \mu\text{m}$
$W_t = 3.5 \mu\text{m}$	

Figure 4.11: Surface Analysis of TiN coated pin at zero degrees

APPENDIX C

appendix A + B removed

SURVEY OF COATING AND DIE CASTING INDUSTRY

C.1 Introduction

Two surveys were sent out to companies in the die casting industry and in the coating industry in order to obtain more information on coatings and their use in die casting. This appendix includes copies of both surveys and the results that were sent back.

C.2 Survey of Die Casting Industry

The following is a list of questions that were included in the survey:

1. What parts do you die cast?
2. What are the typical sizes of your dies? the length between the tie bars?
3. What are typical values for the cavity size in these dies (projected area, volume)?
4. What materials are your dies made of?
5. Approximately for how many shots do your dies last?
6. What level does the cavity pressure reach in your dies during normal operation?
7. What coatings do you use to protect your dies?

8. Who is your coating supplier?
9. What method is used to apply these coatings?
10. What is done to prepare the surface before the coatings are applied?
11. What type of heat treatment do the dies receive as part of the coating process?
12. How thick are the coatings?
13. Approximately how many shots do the coatings last? How many times do the dies get recoated during their lifetime?

Sample responses were:

Ahresty:

1. Automotive parts, primarily using 383 and 393 Al alloys.
2.
 - a) 350 ton machine - 580 mm x 610 mm
 - b) 500 ton machine - 660 mm x 660 mm
 - c) 850 ton machine - 850 mm x 850 mm
 - d) 2250 ton machine - N/A
3. Casting sizes range from 90 mm x 90 mm x 65 mm to 400 mm x 500 mm x 100 mm
4. Premium H13 for cavities, cores, slides and ductile cast iron or 4140 for holding blocks and die bases
5. 80,000 - 150,000 shots
6. ~600 kgf/cm²
7. Nitride, Metal Life, Armoloy, Solvenite
8. Armoloy of Ohio, Badger Metal, and Pro-Long Tool Life Inc.
9. Unknown
10. Cleaned and shotblast

11. Unknown
12. Unknown
13. No accurate information and never recoated.

Doehler-Jarvis:

1. Automotive components, including transmission housings, engine covers, valve covers, etc.
2.
 - a) 800 ton machine - 4 ft. x 4 ft.
 - b) 3500 ton machine - 10 ft. x 10 ft.
3. 153 in³ to 867 in³, 15 to 85 lbs. of Aluminum
4. Premium H13
5. 5000 - 200,000 shots
6. 8 to 15 ksi
7. Melonite, Ferritic Nitrocarburize, CrC
8. Done in house, Multi Arc, Balzers, Dynamic
9. CVD, Salt Baths
10. Degreased
11. Not heat treated
12. 0.0002 - 0.0004 in.
13. 5000 - 100,000 shots

Ford:

1. Alternator housings, throttle bodies, windshield wiper gear housings
2. 29.25 in x 29.25 in

3.
 - a) Alternator housing: 76 in², 47 in³
 - b) Windshield wiper gear housing: 65 in², 40 in³
4. Die blocks are 4140, inserts are H13
5. 80,000 - 150,000 shots
6. 15,000 - 20,000 psi
7. Do not use coatings
8. N/A
9. N/A
10. N/A
11. N/A
12. N/A
13. N/A

GM Powertrain:

1. Automotive transmission cases and V8 engine blocks
2. 60 in. x 60 in.
3. Unknown
4. Premium H13
5. 180,000 shots
6. 8,000 - 10,000 psi
7. Chromium Nitride, Titanium Aluminum Nitride
8. Multi Arc, Gold Star
9. PVD
10. Unknown
11. Parts are heat treated prior to coating to HRC 46
12. Unknown
13. Unknown

Ryobi:

1. Transmission cases, 4 - 50 lbs
2.
 - a) Size of die - 70 in. x 74 in.
 - b) Tie bar length - 60 in.
 - c) 3500 ton machine
3. Pouring weight of 50 - 70 lb, part weight of 30 - 50 lb, 400 - 500 in²
4. H13, SKO-61
5. 180,000 shots
6. Assumed to be 10,000 psi
7. TiN or Nitriding on core pins; inserts and dies are just heat treated
8. Supplier is in Japan
9. PVD for TiN, and salt bath for nitriding
10. Unknown
11. HRC 46-47 for TiN, HRC 58-60 for Nitriding
12. No information
13. No information

C.3 Survey of Coating Suppliers

This survey was sent to approximately 10 coating suppliers and heat treaters.

The following questions were included in the survey:

1. What coatings do you manufacture for use in die casting?
2. What method is used to apply these coatings?
3. What is done to prepare the surface before the coatings are applied?
4. How thick are the coatings once they are applied?
5. What type of heat treatment is necessary with the coating application process?
6. What kind of mechanical property data do you have on your coatings?
7. Do you have information regarding the lives of your coatings? Approximately how many shots will they last in a typical die casting operation?
8. Do you have information regarding the level of cavity pressure that your coatings must withstand in a typical die casting operation?
9. Do you have any suggestions for what factors to investigate, or do you have any overall suggestions for the experiments?

Sample responses:

Arvin TD:

1. Thermal diffused Vanadium Carbide
2. Immersion in salt bath under ambient atmosphere
3. Polish and degrease
4. 5 - 10 micrometers thick
5. Substrate steel hardening
6. Static bending, impact bending, heat checking, fatigue, cracking in carbide coating, bonding strength
7. Yes, thousands to tens of thousands, highly depending on working conditions
8. No concrete data. Successful use for aluminum die casting dies so far, can support the idea that our coating withstands at minimum typical operating conditions
9. According to our experimental results so far, carbide coating by TD did not have cracking, even to ten million cycles, unless the applied stress is large enough to induce plastic deformation of substrate

Balzers:

1.
 - a) CrC for aluminum, especially high silicon alloys
 - b) TiN for zinc and low temp. (<1000°F) casting
2. Ion plating by PVD

3. A clean surface which allows direct deposition of coating on a metal substrate is best. BTC has facilities for vapor honing and degreasing. Other treatments which must be applied are outside the scope of BTC services.
4. TiN is typically 1 - 5 micrometers. CrC is typically 2 - 6 micrometers. Both films can be double coated to increase the thickness if necessary
5. Substrates should be tempered at or above 950°F. Multiple temperings cryogenic treatment per suppliers recommendations aids in developing dimensional stability for close tolerance tooling
6.
 - a) TiN - 2300 Vickers Hardness, Gold color, 1-5 microns thick, cubic crystal structure, 1000°F approx. oxidation resistance, 0.4 - 0.55 approx. coeff. of friction between coating/steel, 5.2 thermal exp. $\times 10^6$ in/in/°F
 - b) CrC - 1850 Vickers Hardness, Silver/Gray color, 2-6 + microns thick, cubic crystal structure, 1250°F approx. oxidation resistance, 0.4 - 0.5 approx. coeff. of friction between coating/steel, 7.2 thermal exp. $\times 10^6$ in/in/°F
7. Typically die life is increased 40 -100% when coating high Si aluminums (Best case to date has been 5x increase when using CrC). Results are similar if not better when using TiN for Zinc die casting.
8. In principle, our coatings must withstand all mechanical loads imposed on the dies in order to stay "on the job." A slight compressive stress, intrinsic to both CrC and TiN, is often advantageous.
9. Will testing be done at room or elevated temperatures? If elevated, will testing be done in air or immersed in molten alloy? Part of the mechanical stress imposed on the die must come from solidification of alloy and contraction of the casting. Another

aspect of mechanical loading is the imposition of stripping forces when castings are removed from cavities. Can bend tests be run with, for example, an aluminum bar against a coated H13 bar?

APPENDIX D

DIE CASTING INDUSTRIES

INDUSTRIAL SURVEY FOR DIE FAILURE AND USE OF COATINGS

SPONSERED BY: THE NORTH AMERICAN DIE CASTING ASSOCIATION (NADCA) AND CENTER FOR DIE CASTING

Please Return to: Dr. Rajiv Shivpuri, Ph: (614) 292 7874 FAX: (614) 292 7852

Dept. Of Industrial, Welding & Systems Engg., The Ohio State University, #1971 Neil Ave., Columbus, OH 43210

I. Part specific data

Note: A sample entry has been made for a Pump Housing, Production Volume -100,000 parts, H13 die steel, Al 380 alloy, Die life uncoated -15,000 shots, Cores coated with TiN and CrC

S.No.	Part Name	Production Volume	Die Steel used #1	Casting Alloy #2	Die failure mechanism(s)#3	Die Life Uncoated	Components coated #4	Die Life Coated			
								Shots	Coating 1#5	Shots	Coating 2#5
e.g.	Pump housing	100,000	a	b	a & b	15,000	cores	50,000	d	33,000	a

Symbols used in the table:

1 (a) H13 (b) H13 premium grade (c) Maraging (d) Others(specify)
 3 (a) Washout (b) Soldering (c) Heat Checking (d) Others(specify)
 5 (a) CrC (b) VC (c) PVD CrN (d) TiN (e) Others(specify)

#2 (a) Al 356 (b) Al 380 (c) Al 390 (d) Others(specify)
 #4 pins, cores, etc. (Please specify)

I. What is the die pre-heat temperature? _____ Method of pre-heat _____

II. Is the die heat treated before or / and after the coating is applied?

☐ Before ☐ After ☐ Before and after

Please specify Heat treatment cycle _____, Hardness _____ Heat Treating company _____

V. Any additional remarks: _____

DIE CASTING INDUSTRIES
INDUSTRIAL SURVEY FOR DIE FAILURE AND USE OF COATINGS
SPONSERED BY: THE NORTH AMERICAN DIE CASTING ASSOCIATION (NADCA) AND CENTER FOR DIE CASTING

Please Return to: Dr. Rajiv Shrivastava, Ph: (614) 292 7874 FAX: (614) 292 7852
 Dept. Of Industrial, Welding & Systems Engg., The Ohio State University, #1971 Neil Ave., Columbus, OH 43210

I. Part specific data

Note: A sample entry has been made for a Pump Housing, Production Volume -100,000 parts, H13 die steel, Al 380 alloy, Die life uncoated -15,000 shots, Cores coated with TiN and CrC

S.No.	Part Name	Production Volume	Die Steel used #1	Casting Alloy #2	Die failure mechanism(s) #3	Die Life Uncoated	Components coated #4	Die Life Coated		
								Shots	Coating #5	Shots
1	Pump housing	100,000	A	B	a & b	15,000	cores	50,000	d	33,000
2	Cylinder Block	100,000	H-13	Al-390	a & b	(uncoated)	Pins, Cores		Nitriding	100,000
3	Transmission Case	180,000	H-13	Al-380	a & b		pins	15,000	Nitriding	15,000
4	Transmission Case	180,000	H-13	Al-380	a & b		pins	6,000	VC	6,000

Symbols used in the table:

#1 (a) H13 (b) H13 premium grade (c) Maraging (d) Others (specify)
 #3 (a) Washout (b) Soldering (c) Heat Checking (d) Others (specify)
 #5 (a) CrC (b) VC (c) PVD CrN (d) TiN (e) Others (specify)

II. What is the die pre-heat temperature? N/A Method of pre-heat N/A

III. Is the die heat treated before or / and after the coating is applied?

[] Before [] After [X] Before and after

Please specify Heat treatment cycle Hardness 46-48 Heat treating company Ill: amca

IV. Any additional remarks:

APPENDIX E

APPENDIX E

Project Meetings

- Revised and finalized project schedules, tasks and presented at the NADCA Die Materials Committee meeting at Cleveland (OH) on Feb 22, 1995
- Meeting with Balzers Inc. at N.Tonawanda(NY) on candidate coatings for production tests
- Meeting with Multi-Arc Inc. at Columbus (OH) regarding application of coatings to die inserts
- Project progress presented at the NADCA Die Materials meeting, Cleveland (OH) , June 1, 1995
- Visit to Ganton Technologies, Racine(WI) on September 11, 1995 to finalize their possible involvement in the project. Ganton agreed to provide the necessary test bed for production trials.
- Progress presented at the NADCA Die Materials Committee meeting at Cleveland (OH), September 13, 1995
- Proposed research plan for 1996-98 presented at the NADCA R&D Committee meeting at Chicago(IL), September 27,1995
- Project progress presented at the DOE workshop on die casting projects at Chicago(IL), September 28, 1995
- Meeting with Multi-Arc Inc. at Rockaway(NJ) to finalize the plan for inclusion of their coatings in the NADCA research, October 6, 1995
- Meeting at Doelher-Jarvis on October 27, 1995 to prepare a final draft of the two page proposal submitted to the NADCA R&D Committee on November 8, 1995
- Meeting at Doelher-Jarvis, Pottstown to discuss their involvement in the project and test die identification on November, 1995
- Visit to Ganton Technologies, Racine(WI) on October, 1995 to study the production parameters and discuss suitable test schemes
- Meeting with Ganton Technologies on December 22, 1995 at Columbus(OH) to discuss and finalize the experimental scheme developed by OSU.
- Project progress presented at the NADCA R&D Committee meeting at Chicago (IL), February 7, 1996
- Meeting with Pace Industries, Monroe City on March 15, 1996 for on-site evaluation of the first batch of production tested pins.
- Project progress presented at the NADCA R&D Committee meeting at ORNL, June 13, 1996.
- Meeting with Multi-Arc Inc. at Columbus(OH) on September 28, 1996.
- Project progress presented at the NADCA R&D Committee meeting at Chicago, October 16, 1996.
- NADCA Coating and Surface Treatment Subtask Group meeting, Feb 20, 1997 at OSU.