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**EFFECT OF COMPOSITION AND PROCESSING
ON THE THERMAL FATIGUE AND TOUGHNESS
OF HIGH PERFORMANCE DIE STEELS**

Final Report

**J. F. Wallace
Y. Wang
D. Schwam**

June 1997

Work Performed Under Contract No. FC07-95ID13360

**For
U.S. Department of Energy
Assistant Secretary for
Energy Efficiency and Renewable Energy
Washington, DC**

**By
Case Western Reserve University
Cleveland, OH**

MASTER

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Mr. Mr. Mark Rodney of Latrobe Steel
Mr. Herb Brucher of Doehler Jarvis
Mr. Corwin Berger of Thausig Associates
Mr. John Fitzgerald of FPM Heat Treating,
Mr. Carl Dorsh of Crucible Research
Mr. Derek Cocks of Eastern Alloys
Dr. Walter Smith of DCD Technologies
Mr. Guy Brada of Finkl and Sons Inc.

Also participating in the activities of the Industrial Monitoring Committee were Dr. Steve Midson and Mr. Steve Udvardy, former and present NADCA Directors of Research.

Numerous companies have contributed to this project with in-kind products and services. Among the main contributors are:

Latrobe Steel with steels for samples and inserts
Alloy Tool Steel with steels for samples and inserts
Uddeholm with steels for samples and inserts
Finkl & Sons Inc. with steels for samples and inserts
Universal Heat Treating with heat treating services
FPM Heat Treating with heat treating services
Lindberg Heat Treating with heat treating services
DCD Technologies with machining and EDM services
MMC with EDM services
ITT Automotive with in-plant evaluation
Imperial Die Casting with in-plant evaluation
General Die Casters with in-plant evaluation

The contributions of these companies and the individuals representing them is gratefully acknowledged.

ABSTRACT

Dies cost about 20% of the total cost of producing aluminum die castings. Improvements in die life could result in annual savings of over \$200 million in US with only a 20% life extension. The objective of this study was to obtain this improvement in average die life by optimizing die steel composition and the die processing. Four different steels, K, Q, C and Premium Grade H-13 have been investigated for thermal fatigue resistance and toughness. Premium Grade H-13 is now the standard die steel for aluminum die castings used in US. K and Q are lower in vanadium content and higher in molybdenum than conventional H-13 die steel. K also has a lower silicon content that should presumably minimize interdendritic segregation. Optimum heat treatment processing has been determined for each steel with respect to austenitizing temperature and tempering conditions. The effect of the quenching rate on the thermal fatigue resistance and toughness of the die steels and the effect of Electro-Discharge Machining (EDM) on the thermal fatigue resistance were also determined. The immersion thermal fatigue specimen developed at CWRU was used to determine the thermal fatigue resistance as characterized by the two parameters of average maximum crack length and total crack area. The Charpy V-notch impact test was used over a -100°F to 450°F testing temperature range to evaluate the toughness and the brittle-ductile transition behavior.

K steel has been identified as superior in performance compared to Premium Grade H-13. Q and C provide lower toughness and thermal fatigue resistance than H-13. Faster cooling rates provide higher thermal fatigue resistance and toughness. Higher austenitizing temperatures such as 1925°F compared to 1875°F provide better thermal fatigue resistance, but lower austenitizing temperatures of 1875°F provide better toughness. Higher hardness improves thermal fatigue resistance, but reduces toughness. A minimum of Rc 46 hardness is desired for aluminum die casting dies. EDM reduces the thermal fatigue resistance compared to conventional machining operations. When the EDM process of multiple small steps of decreasing energy and post-EDM treatments are employed, the effect can be reduced to a very slight amount.

Preliminary evidence of the superior performance of the K steel has been provided by ongoing field testing of inserts in multiple cavity dies.

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EFFECT OF COMPOSITION AND PROCESSING ON THE THERMAL FATIGUE AND TOUGHNESS OF HIGH PERFORMANCE DIE STEELS

I. INTRODUCTION

Die casting is an economical way to produce large quantities of complicated-shaped products of light metals with high precision⁽¹⁾. In the process, molten metal is injected under high velocity into a water cooled metallic mold or die. It solidifies as a net shape product⁽²⁻³⁾. When solidification is completed and the die casting has cooled sufficiently, the die is opened and the part is ejected by mechanically activated ejector pins. The die is subsequently exposed to air, sprayed with a lubricant, closed and molten metal is again injected into the die to complete the cycle. The major economic advantages of the process is that it can produce castings with close dimensional tolerances, smooth surface finishes and fine, intricate details at high production rates.

However, due to thermal and mechanical cyclic loading and reactions with entering molten metal, die casting dies are subject to severe damage including erosion, chemical attack, gross cracking and thermal cracking⁽⁴⁻¹⁴⁾. Damage to the dies increases production costs and reduces casting quality. With the increasing amount of die casting of aluminum, the damage to the dies becomes a serious problem. The advantages of the die casting are offset to some extent by the high cost

of the die depending on its intricacy and complexity, the die can even cost more than the machinery used to operate it ^(5,6).

Die casting with aluminum alloys requires die steels with higher strength and toughness than that used for lower melting temperature metals. This steel is both more expensive and more difficult to machine. In aluminum die casting, typical die life ranges from 100,000 to 150,000 castings ^(15,16). It costs about 20% of the total cost of producing aluminum die castings. As the number of parts that can be cast from a single die increase, the process becomes more economical. Additional costs are incurred when the production of castings is interrupted or die failure necessitates repairs. Therefore, increasing the production life of a die through die steel improvement and die failure prevention can make a major contribution toward the profitability of the die casting operations. Improvements in die life could result in annual savings of over \$200 millions in US with only a 20% life extension. This is based on the estimated annual die production of one billion dollars in US. The objective of this study was to obtain this improvement in average die life by optimizing die steel composition and the die processing.

The major tasks of the project were:

- Evaluate new die steel compositions that have been developed for demanding applications and compare them to Premium Grade H-13 die steel to find the best steel available for aluminum die casting dies.

- Optimize the austenitizing treatment of the new composition steels. Assess the effects of fast, medium and slow cooling rates during heat treatment, on the thermal fatigue resistance and toughness of the die steels.
- Determine the effect of Electro-Discharge Machining (EDM) on the thermal fatigue resistance of the steels.

The immersion thermal fatigue specimen developed at CWRU was used to determine the thermal fatigue resistance as characterized by the two parameters of average maximum crack length and total crack area. The Charpy V-notch impact test was used over a -100°F to 450°F testing temperature range to evaluate the toughness and the brittle-ductile transition behavior.

II. PROPERTIES OF DIES AND DIE STEELS

2.1. Die Failure Modes

Failure modes identified for aluminum die casting dies are mechanical erosion, chemical attack, gross cracking and thermal fatigue cracking. Two types of mechanical erosion have been defined^(5,6,11-15). Wear erosion is caused by the abrasive action of the molten metal as it flows over the die surface. Cavitation erosion is the result of the collapse of vapor-filled bubbles in the molten alloy near the die surface. This causes high pressures at the surface, resulting in stress induced strain at the die surface^(6,12). The repetitive straining of the surface is a source of both mechanically and thermally induced fatigue.

The die surface is attacked by a number of ways, first, through compositional changes in the die surface and by the adhesion or welding of the cast metal to the die surface. This latter behavior is known as soldering and results in damage to both the cast part and the die. Compositional changes in the steel at the die surface have the effect of degrading the strength and ductility of the die surfaces and result in accelerated thermal fatigue failure⁽¹²⁾. Chemical attack occurs through compositional changes in the die surface by means of decarburization of steel dies. Decarburization is the loss of carbon at the die surface during heat treatment or production use and results in a lower surface hardness.

The surface of the die is also tempered by its cyclic exposure to higher temperatures. This tempering plus decarburization can reduce the hardness of H-13 die steel die to about 35 Rockwell C while the die interior has an optimum hardness of 46-48 Rockwell C^(6,9-11). Because the surface has a strength below that needed for the die casting operation, early heat checking may occur. Oxidation or corrosion is also related to heat checking during both crack initiation and crack propagation stages.

Gross cracking is catastrophic in nature and not as directly related to the surface of the die cavities. It is produced by thermal and mechanical stresses on the die. It may require that the die be replaced because of extensive or complete cracking⁽⁸⁾. These cracks frequently occur at low temperatures or in die steels with low fracture toughness. They also initiate from deep heat checks. Thermal stresses result from temperature variations in the die produced by heating or cooling. This thermal stressing of the die with mechanical stressing may exceed the die fracture strength^(6,11). This cause can be minimized by avoiding high thermal and mechanical stresses and by increasing the fracture toughness of the die material.

Heat checking, a result of thermal fatigue, is the most frequent cause of the failure of aluminum die casting dies. Thermal fatigue is caused by the cyclical temperature fluctuations present in the die casting process. It induces the stress and strain conditions that occur when the thermal expansion or contraction of a section is restrained by the surrounding material. When such local stresses exceed the elastic

limit or yield strength of the die, plastic deformation will occur, and with sufficient cycling, microcracks will nucleate. A repeated series of heating and cooling of the surface can produce a fine network of cracks in the die surface. This is recognized as heat checking which is also called thermal fatigue cracking or craze cracking. The cracking starts after a number of die casting cycles (initiation) and grows at an accelerated rate (propagation). Cracks initiate and propagate in both an intra- and inter-granular manner and are filled with oxides⁽¹³⁾.

Corrosion is also involved in the initiation and propagation of heat checks. The die is exposed to an oxidizing atmosphere which can cause corrosion pits that are filled with oxides. Because the oxides occupy a large volume than the metal that was previously present, a wedging action occurs which contributes to crack propagation^(11,17). From practical viewpoint, die failure occurs after a period of time. The die is normally removed from service after the heat checks have grown to a critical length or when the cosmetic appearance of the die casting is not acceptable, or when "sticking" of the cast part to the die surface occurs.

2.2. Thermal Stresses

The thermal stresses that occur in the die develop from the thermal gradient across the die section. The thermal gradient forms as a result of the heating and cooling of the surface during the injecting, ejecting and lubricant spraying stages of

the casting cycle. When the molten aluminum is injected into the die, the die surface heats up considerably compared to the cooler underlying mass of the die. This sets up an initially steep thermal gradient. As a result, the surface expands more than the interior and because the interior is more massive, it prevents the surface from expanding. As a result of this restraint, the surface is placed under compression. As the increase in temperature lowers the yield strength of the material, the compressive strains may become plastic. The surface temperature decreases rapidly as heat is conducted into the lower layers. When the casting is ejected, cooling to the surroundings from the surface and the spray of die lubricants further decrease the surface temperature⁽¹⁸⁾. If the surface cools more rapidly than the interior, the compressive strains are relieved and tensile strains may be created.

Figure 2.1 shows schematic diagrams of the temperature, stress and strain distributions experienced by casting dies both on heating (during casting) and cooling (after the casting has been extracted from the die)⁽¹⁹⁾. Each half of the casting cycle is portrayed by three schematic drawings. Figure 2.1(a) through 2.1(c) show the effect of increased surface temperature on the stress and strain distribution from surface to interior of the die material. Figure 2.1(d) through 2.1(f) illustrate the subsequent stress and strain conditions which exist during the second or cooling period of the casting cycle. It becomes clear that the thermal gradient is a function of the thermal conductivity of the die material. Increased thermal conductivity will result in a lower

Figure 2.1. Temperature, Stress and Strain Distributions when Dies are Exposed to Molten Metal⁽¹⁹⁾

thermal gradient and thus lower stresses and strains. During heating of the injection half of the die casting cycle, the surface attempts to expand putting it in compression with respect to the die interior, as shown in Figure 2.1(b). The corresponding compression strain is shown in Figure 2.1(c). The combination of the temperature gradient and the coefficient of thermal expansion of the die material determines the magnitude of the die surface stress. Initially, the surface deformation (strain) is within the elastic capabilities of the die material. However, if the combination of the temperature gradient and the prevailing thermal expansion are high enough, the compression stress developed will exceed the elastic limit of the die material as can be seen in Figure 2.1(b), by the change in slope of the stress plot near the die surface.

Plastic deformation will take place after the initial elastic strain has occurred. As the increase in temperature lowers the yield strength of the material, the compressive strains becoming plastic will be enhanced by the high operating temperature. The elevated die surface temperature T_{s1} declines during part ejection and reaches some minimum value T_{s2} . T_{s2} varies widely with specific die casting practice. Figure 2.1(d) shows T_{s2} at a lower temperature than the mean die temperature T_m . This surface temperature could go down well below the mean die temperature, T_m , depending on the type and quantity of die release used and the time the die cavity is open, etc. In the discussion of thermal fatigue, the actual value of the temperatures, within limits, is less important than the difference between them, since the magnitude of the temperature change $\Delta T = T_{s1} - T_{s2}$ is directly related to the

maximum stress and strain that the die surface undergoes. As the surface metal cools down with respect to adjacent metal further into the die block it attempts to contract more rapidly than the warmer adjacent metal and put in tension. If the thermal stress is larger than the elastic limit of the material in its prevalent condition, reversed plastic deformation will occur. Exceeding the elastic limit by even a small amount will ultimately result in a fatigue failure after a sufficient number of cycles has occurred. The inversion shown in both the stress and strain in Figure 2.1(e) and (f) result from the change in direction in the temperature profile shown in Figure 2.1(d).

The thermal stresses that occur in the die develop from the thermal gradient across the die section and are determined by the combination of the temperature gradient and the thermal expansion as just discussed. For a simple case of a bar with fixed end supports, the thermal stress generated by a temperature change ΔT is:

$$\sigma = \alpha E \Delta T \quad (2-1)$$

where α is the linear thermal coefficient of expansion and E is the elastic modulus⁽²⁰⁾.

When the continued cycle of alternating stresses exceeds the material fatigue strength, heat checks form. For biaxial condition the stress can be given by:

$$\sigma = \frac{\alpha E}{1 - \nu} \Delta T \quad (2-2)$$

where ν is Poisson's ratio.

A rough estimation of the order of magnitude of thermal stresses encountered by the die surface can be made by the simple calculation using Equation (2-2):

Assuming maximum die surface temperatures of $1100^{\circ}\text{F}^{(8,13)}$ and die preheating temperatures of 500°F for aluminum and substituting the typical values of die steels^(8, 21):

$$\begin{aligned}\sigma &= \frac{8 \times 10^{-6}}{1 - 0.3} \times 2.5 \times 10^7 (1100 - 500) \\ &= 171,429\text{psi} \approx 171\text{ksi}\end{aligned}$$

where $\alpha = 8 \times 10^{-6} \text{ in/in}^{\circ}\text{F}$

$$E = 2.5 \times 10^7 \text{ psi}$$

$$\nu = 0.3$$

Since the yield strength of H-13 steels (oil quenched from 1850°F and double tempered to Rc 48 hardness) is approximately 140 ksi at 1000°F , these severe thermal stresses at elevated temperature can produce plastic deformation. However, such high thermal stresses never fully develop because α , E , ν and the thermal conductivity all change with temperature⁽²¹⁾. More detailed work by Graham who carried out a finite element analysis indicated that the maximum compressive stress may well remain below the yield strength of H-13 steels for the aluminum die casting⁽²²⁾.

2.3. Thermal Fatigue Resistance

The thermal fatigue resistance of die materials is greatly influenced by a number of material properties. The more important of these are the coefficient of thermal expansion α , the thermal conductivity k , and the resistance of the material to an alternating strain $\pm\varepsilon=\alpha\Delta T$. When thermal strains are a result of a material being constrained, the thermal conductivity k will be of no consequence; on the other hand, when thermal strains are a consequence of thermal gradients resulting from rapid heating or cooling, a high conductivity may reduce the temperature gradients and hence the thermal strains. This condition exists when the rate of heating is not so high that the outside edges of the specimen reach their maximum temperature before any significant temperature change can occur in the body of the material. Thus, depending on operating conditions, either of the parameters ε/α or $k\varepsilon/\alpha$ can be used as an indication of thermal fatigue resistance. The parameter $k\varepsilon/\alpha$ is applied to the die casting condition and defined here as K .

$$K = k\varepsilon / \alpha \quad (2-3)$$

ε can be given by the modulus E and fatigue strength at the mean temperature σ_f :

$$\varepsilon = \sigma_f / E \quad (2-4)$$

So the thermal fatigue resistance of a die steel can be given by:

$$K = \frac{\sigma_t k}{\alpha E} \quad (2-5)$$

A high value of K indicates good thermal fatigue resistance. The strength of a die steel at elevated temperatures should be high because the yield strength tends to decrease with increasing temperatures. As a result, lower stresses are needed for plastic deformation to occur, subsequently causing upsetting failure by means of heat checking. High thermal conductivity will reduce thermal fatigue by establishing a low thermal gradient between the surface and the underlying layers. Equation (2-1) and (2-2) indicate that a lower temperature gradient will decrease the stresses in the die and therefore lower the tendency for cracking. Equation (2-1) and (2-2) also shows that lower value of the modulus of elasticity and the coefficient of thermal expansion will produce lower stresses in the die. A lower modulus results in lower stresses at a given strain level as shown by equation (2-4). Since the different surface layers are at various temperatures, differences in relative expansion between the surface and subsurface result, thereby increasing the tendency for cracking. Lower values of the coefficient of thermal expansion will reduce the amount of relative expansion between the surface and the underlying layers. Higher thermal conductivity also reduces the thermal gradient and relative expansion between layers of the die.

High hot hardness is also desirable because it prevents softening of the surface. At higher temperatures, the surface loses strength and hardness which lowers

the thermal fatigue resistance. However, too high a hardness will reduce the toughness of the material and this can lead to gross cracking. In selecting the optimum die material, a compromise is necessary optimizing the range of properties.

2.4. Thermal Fatigue Evaluation and Test Techniques

Most of the thermal fatigue tests impose more severe conditions than actual production dies would experience. Tests are accelerated to reduce the testing times to reasonable experimental interval. In all the cases, the thermal fatigue or heat checking is evaluated by examining and comparing the cracks developed on the tested specimen at the severe thermal stress concentration points after a certain number of thermal cycles. The first thorough examination of thermal cycling was performed in the middle 1950's by Northcott and Baron⁽²³⁾. They developed a wedged-shaped thermal fatigue specimen, which was heated by a high frequency induction coil and cooled by a water-cooled copper block. After a certain number of cycles, the sample was removed, sectioned, and microscopically examined.

Glenny and Taylor⁽²⁴⁾ first used a fluidized bed system for heating tapered disc samples in 1959. A 1.625" in diameter by 0.275" in thickness disc sample was tapered to a point at the edge. In 1969, Rostocker⁽²⁵⁾ expanded on Glenny and Taylor's idea by utilizing fluidized beds for both heating and cooling 2-1/4" in diameter by 1/8" in thickness disc samples. Instead making the edge tapered discs, K. Hayashida⁽²⁶⁾

recently made a serial of 0.5 mm radius notches, acting as stress concentration points, along the edge of the thin disc samples to enhance the test condition and eliminate making the sharper edges.

In the middle 1960's, Wollering and Oertle⁽²⁷⁾ studied heat checking with a rotating specimen holder and a oxy-acetylene flame as the heat source. Immediately following with the heat cycle (4-5 seconds), samples were rotated into a water bath for cooling. This automated test left concentric rings due to the circular nature of the flame, thus indicating the temperature distribution on the surface. The sample dimensions for this investigation were 1" x 1" x 1-1/2". Results from this test were similar to those from the wedge test.

Resistance heating was first utilized by Korostolev and Kunilovsii⁽²⁸⁾ to investigate heat checking and die life in the middle 1970's. Advantages of this type of test include small sample size and the ability to control heating and thermal gradients. However, an external constraint was utilized rather than an internal constraint. In addition, localized overheating of the crack tip caused by a buildup of current tended to exaggerated results.

High-frequency (HF) induction heating of small scale samples has been performed by both Holmes and McClintock⁽²⁹⁾ using disc-shaped samples and by Malm, et al.⁽³⁰⁾ using cylindrical shaped samples. After the disc-shaped samples were heated, cooling was performed by forced air. Holmel et al.⁽³¹⁾ performed thermo-

elastic finite element analysis on the discs to predict the stress-strain behavior of the material during the thermal cycling. Since the disc-shaped samples were so thin in this test, the constraint was not the same as that experienced by dies.

Malm, et al.⁽³⁰⁾, from Uddeholms AB, used high frequency (HF) induction heating of 10 mm in diameter by 20 mm long cylinders for an industrial test to determine heat checking susceptibility of various hot work die materials. A flat surface of five millimeters wide was ground along the length of the sample, so that heat checks would initiate and grow in this area, and microscopic examination could be performed after a certain number of cycles. After heating, the sample was then water sprayed for quenching. A temperature gradient and constraint were created similar to that experienced by dies and molds. Geller, et al.⁽³²⁾ also used HF induction heating to characterize heat checking in various hot work materials. P. Hochanadel⁽³³⁾ recently used the Uddeholms thermal fatigue tester to test the thermal fatigue behavior of investment cast H-13 steel.

The immersion thermal fatigue tester developed at CWRU more closely simulates the environment that the aluminum die casting dies encounter. It has been used for thirty years to evaluate a large number of potential die materials for aluminum die casting dies⁽¹³⁾. A 2" x 2" x 7" rectangular parallelepiped specimen with a 1-1/2 inch diameter 6-1/2 inch deep hole in the center for internal water cooling is dipped into a molten aluminum bath and systematically extracted and cooled for a number of cycles followed by the examination and characterization of the thermal

fatigue cracks formed on the specimen during the thermal cycling. The test produces the considerable constraint and high thermal fluctuations during the cycling. It will be discussed in detail in Chapter IV.

2.5. Effect of Metallurgical Variables on the Die Life

The metallurgical variables have great influence on the die life of a die steel through the effects on both thermal fatigue and fracture toughness of the steel. It has been established that the austenitizing temperature is an important metallurgical variable^(8,11,34-37). When the austenitizing temperature is increased from the conventional hardening temperature of 1800°F upward to 2200°F for H-13 steel, a steady improvement in the resistance to heat checking occurs. This improvement is attributed to the greater resistance to tempering during casting because of the increased solution of alloy carbides. However, above 2000°F, significant grain growth occurs and reduced amounts of carbides are available for the inhibition of grain growth. The Charpy impact values and the fracture toughness decrease rapidly as the austenitizing temperature is raised to above 2000°F^(8,34,37). Considering both the thermal fatigue and fracture toughness behavior, a 1900°F temperature may well be the best austenitizing temperature for H-13 steel. The optimum microstructure for the die steel is a fine martensitic structure obtained with rapid cooling or a completely bainitic structure⁽³⁴⁾. An optimum hardness level is obtained by quenching and

tempering. The higher hardnesses over Rockwell C 50 are too brittle with lower fracture toughness, the lower hardness lacks sufficient strength and reduces the thermal fatigue resistance.

Since the level of strength and toughness governs thermal fatigue behavior, any change in local chemistry during heat treatment either through decarburization or carburization may affect thermal fatigue behavior. Therefore, the carbon content of the steel at the surface and its associated hardness level has a marked effect on thermal fatigue life. Accordingly, decarburization is detrimental and causes premature heat checks because the low carbon layer has a lower strength^(7,9,22,38). On the other hand, significant carburization is also detrimental and exhibits a lower heat checking resistance because the surface layer does not have enough toughness^(38,39). However, it was reported that some degree of carburization can increase thermal fatigue life⁽²²⁾. Therefore, it is concluded that decarburization and heavy carburization cause premature heat checking and slight carburization may have little effect⁽³⁴⁾.

Thin hard nitride and carbide coating techniques have been developed and are used in die casting dies⁽⁴⁰⁾. The coated layer has a thickness less than 10 or 20 μm with extremely high hardness, HV2000-3000. It is claimed that the thin hard coating process, CVD(Chemical Vapor Deposition), PVD(Physical Vapor Deposition), PCVD(Plasma Chemical Vapor Deposition) and TRD(Thermal-Reactive Deposition and Diffusion), can provide remarkable improvement of the surface properties of steels highly associated with damage on die casting dies. Many new methods for

producing unique modifications of material surfaces have been developed in the past 3 decades⁽⁴¹⁾. However, research for evaluation of coated steels is not been developed sufficiently to analyze its effect on the die life.

Nonmetallic inclusions are generally considered undesirable constituents and are usually required to be held to a minimum within practical limits⁽³⁸⁾. The use of premium grade H-13 steel provided by VAR and ESR previously has resulted in a significant improvement of heat checking resistance.

Die steels may have different structures depending on the cooling rate from the austenitizing temperature⁽⁴²⁾. The final microstructure should be a uniform tempered martensite without carbides precipitated at grain boundaries and undissolved carbides. If the steel is allowed to cool slowly during hardening, the thermal fatigue life and Charpy impact energy values are reduced when heavy carbide precipitation occurs at the grain boundaries^(34,36,42).

An idealized microstructure should consist of tempered martensite within which are small, rounded, and evenly dispersed pro-eutectoid carbides and with a finer scale carbide dispersion within the martensite product itself. Such a structure can be obtained if the prior annealed structure is such that all the precipitated and primary alloy carbides are evenly distributed within the matrix. This means that the prior structure should be homogeneous and free of carbide stringers and severe alloy banding. The widely used specifications to ensure high homogeneity of die steels

based on annealed microstructure ratings are NADCA #207-90 and Chrysler NP-2080^(43,44). These annealed structure ratings are primarily based on the homogeneity of carbide distribution which directly relate to the impact toughness and thermal fatigue resistance of the die steels^(45,46). A good annealed structure provides about 14 ft-lb room temperature impact toughness after it is austenitized at 1875°F and double tempered to 44-46 Rc hardness. A poor prior structure has only half the value under the same heat treatment⁽⁴⁵⁾. A new acceptance standard for die steels has been developed and recently suggested by B. Gehricke et al.⁽⁴⁷⁾.

Dies for aluminum die casting require high thermal fatigue resistance and toughness properties of the die steels. Carbide precipitation on grain boundaries can reduce the properties⁽⁴⁸⁾. The heat treatment may not totally suppress the existence of carbide precipitation, so changes of the alloy composition can be employed to reduce the carbide content and the intensity of carbide precipitation on grain boundaries. Based on H-13 die steel, the basic carbide forming elements are carbon, chromium, molybdenum and vanadium. One of the objectives of the project is to optimize the steel composition for better die steels.

2.6. Effect of Cooling Rates

Cooling rate has a great influence on the properties of the die steels. If the cooling rate is too slow, upper bainitic or even pearlitic transformation from austenite

may occur. The tempered upper bainitic or pearlitic structures result in significantly poorer than those of tempered martensite. This result is from the presence of interlath MC carbide platelets, which may form during the slow quench, and to the presence of M_2C and $M_{23}C_6$ carbides, which form during tempering. Therefore, a rapid cooling rate from the austenitizing temperature provides a fully martensitic structure. It has also been determined ⁽³⁶⁾ that these bainitic structures can be prone to a 1100°F tempering embrittlement regardless of the austenitizing temperature employed. The cause of this embrittlement, however, was not adequately addressed. It was also shown that cooling too slowly from the austenitizing temperature produces intergranular carbides, which are detrimental to the properties^(49,50). The cooling rate in the temperature range 1750°F to 550°F should be at least 50°F/min to avoid excessive intergranular carbide precipitation^(49,50).

A previous project conducted at CWRU has demonstrated⁽⁵⁰⁾, as shown in Figure 2-2, that cooling rates of about 50°F/minute from the austenitizing temperature produced fairly high although not the best impact resistance and a tempered bainite structure with some grain boundary carbides. The bainitic matrix structure with some grain boundary carbides obtained by these intermediate cooling rates may be capable of producing long lasting dies without requiring the faster cooling rate needed to obtain a martensitic structure. This slower cooling rate is more attainable in vacuum furnaces and avoids much of the distortion and heat cracking that could occur at faster rates of cooling.

Figure 2.2. Effect of Cooling Rate on Toughness of H-13 at 46 Rc

However, the previous studies had shown that the optimum combination of properties was only obtained by more rapid cooling rates that assured the rapid cooling to martensite. This leaves unanswered the question about the thermal fatigue properties of these bainitic structures. One task of the study was designed to provide the answer to this question

2.7. Effect of Electro-Discharged Machining(EDM)

At present time, two procedures are usually employed for die steel dies for aluminum die castings. The first is to machine the block of die steel close to final dimensions in the annealed condition when the steel is relatively soft (about 26 Rockwell C hardness). Then the die is heat treated by austenitizing in a vacuum furnace, cooled as rapidly as feasible by nitrogen gas blown into the vacuum furnace at pressures that are several times atmospheric and then double or triple tempered to hardness required hardness levels, 45-47 Rockwell C for H13 die steel. The distortion that occurs during this heat treatment is corrected by final machining. Since these final machining operations are conducted on hardened steel, they are slow and expensive. It is desired to keep these operations to a minimum by predicting and controlling the distortion. The amount and causes of this distortion when heat treating and cooling H13 steel dies in vacuum furnace is being studied by D. Schwam, R. Aikin and J. Wallace at CWRU⁽⁵¹⁾. The research is to determine the extent of this

distortion in various shaped dies at different rates of gas cooling . This will permit the machining of the die in the annealed state to as close to final dimensions as feasible, thereby reducing final machining operations.

The second method of processing dies involved the use of the EDM procedure. This method is to block out the general dimensions of the die by machining the annealed block of steel. Generous allowances are made for compensating for distortion after heat treatment. Since final finishing by EDM can be accomplished as readily in the hardened as in the annealed state, the blocked out die can be heat treated to its high hardness level in conventional atmospheric controlled furnaces. Sufficient additional finish is allowed to remove the dimensional variances from distortion and any changes in surface conditions. The rough machined die, before EDM finishing, can be cooled considerably faster from the austenitizing temperature than is possible by gas injected into a vacuum furnace. The use of oil quenching may be employed and the optimum microstructure of the die can be achieved. The additional finish on the die configuration allows for more distortion to be removed than feasible with the conventional machining procedures employed for the dies heat treated in vacuum furnaces.

EDM is becoming more and more widely used in die manufacturing and is reported to be used for about one half of modern dies. This process is capable of producing fine intricate details such as holes with diameters as small as 0.002 inch⁽⁵²⁾. Because the process is independent of work piece hardness, it has the ability to

machine fully hardened tool steels without the use of hard tools. This minimizes or eliminates adjustments due to any associated distortions. While conventional machining processes require hard carbide tools, the electrodes used in EDM are commonly made from graphite or copper which are soft and easier to machine⁽⁵³⁾.

Metal removal produced in EDM occurs by a series of electrical spark discharges. The process affects the microstructure of the surface layers of the work piece^(8,54-57). In die steels, four layers form in the EDM surface as shown in Figure 2.3: a) the recast layer, b) the untempered martensitic layer, c) the tempered layer, and d) the unaffected parent material. The recast layer is present as a brittle, non-etchable white layer containing cracks^(36,39,43,54,55,58). This is the material that was melted and rapidly resolidified on the surface and not flushed away by the dielectric fluid. This layer is infiltrated with carbon which can originate from a graphite electrode and from hydrocarbons that form from the dielectric when it breaks down^(39,42,55,58,59). Depending on the machining parameters and the work piece material, the white layer may range in thickness from 5 to 15 microns. The zone immediately under the white layer has been heated into the austenitizing temperature range by the heat produced during EDM and rapidly cooled by conducting the heat into the body of the die. During the rapid cooling from this temperature, hard brittle untempered martensite is formed. In the regions further below the surface, the temperatures attained are lower and in the tempering temperature range. The resulting steel in this layer is softer than the original hardness obtained by heat treatment. The tempered zone has a thickness

The diagram shows a cross-section of the surface layers of H-13 steel after EDM. The top surface is irregular and jagged. Below it are four distinct layers, each with a label and corresponding depth and hardness values.

	DEPTH (um)	HARDNESS (Rc)
MELTED AND SOLIDIFIED	5 - 25	56 - 58
UNTEMPERED MARTENSITE	15 - 40	50 - 54
TEMPERED LAYER	30 - 85	34 - 43
UNAFFECTED BASE METAL		46 - 48

Figure 2.3. Surface Layers on EDMed Surface of H-13 Steel

that can reach up to 85 microns^(56,57). When the EDM process is properly conducted, the untempered martensitic layer is softened by tempering at temperature about 50°F below the highest tempering temperature used for the die. The tempered layer can not be rehardened, so it remains in the EDM processed die^(62,64).

Although EDM does not exert mechanical forces on the work piece, it is evident that residual stresses exist in EDM surfaces. High tensile stresses have been measured at the surface. These stresses fall rapidly with increasing depth to a relatively low value and then change at further depth to small compressive stresses^(36,60). The stresses arise mainly as a result of the thermal contraction of resolidified metal or recast layer onto the parent metal^(42,60). This induces plastic deformation and tensile stresses. The thermal cycle at the surface causes steep temperature gradient normal to the surface in a somewhat similar manner as thermal conditions that occur at the die surface during the casting process. This can contribute to the development of residual stresses in the die^(60,61). However, these stresses are reduced coincidentally by the tempering treatment of the untempered martensitic layer.

III. POTENTIAL NEW DIE STEELS

Some new die steels have been reported to provide improved die life for die casting dies^(26,74,75). The amount of the important alloy elements in the composition of these steels is shown in Table 3-1. When compared to premium grade H-13, K and Q steels are lower in silicon, vanadium and chromium and higher in molybdenum. C steel is lower in carbon, molybdenum and vanadium than H-13 but much higher in chromium.

These steels were heat treated in a similar manner as premium grade H-13: rapid cooling from a high austenitizing temperature with a double or triple temper. Some of these steels, when a high austenitizing temperatures of 1900°F is employed, are significantly higher than for H-13 steel in resistance to tempering. The resistance to tempering is an important consideration in reducing thermal fatigue cracking. The surface of H-13 dies that have suffered severe thermal fatigue cracking have always been softened below 40 Rockwell C hardness by this tempering action.

Table 3-1. The composition of various steels

wt %	P.G. H-13	K	Q	C
C	0.40	0.38	0.37	0.26
Si	1.00	0.21	0.30	0.85
Mn	0.35	0.42	0.63	0.90
P	0.025	0.01	0.015	0.019
S	0.001	0.002	0.001	0.001
Cr	5.25	5.20	2.46	10.94
Mo	1.50	1.85	2.22	0.90
V	1.00	0.51	0.84	0.50
Cu	0.08	0.03	0.22	0.05
Ni	0.15	0.03		0.95
W		0.01		0.90

Die steels of the general K composition have been tested for thermal fatigue cracking by a severe test that involves heating and quenching in water. While this test may not be as directly relevant to die casting dies as the immersion test described elsewhere in this thesis, it shows that the new composition has twice the heat checking resistance as H-13. It is noted that the results of the heat checking tests show better resistance at higher hardness for both H-13 and K. It is also noted that this steel with its lower silicon and vanadium and higher molybdenum contents are used at hardness up to 50-52 Rockwell C.

The use of the new steel at the higher hardness may provide a partial rationalization for its success. It has been shown in previous work conducted at CWRU⁽⁶⁴⁾ that the resistance to thermal fatigue cracking improved by hardnesses up to Rockwell C 50. However, the significant loss in toughness that occurred at these higher hardness with H13 did not permit the dies to be used at this hardness level. The reported Charpy V-notch impact energies at room temperature for K at 52 Rockwell C hardness are slightly higher than for H-13 steel at 46 Rockwell C hardness. This indicates that K can probably be employed at the higher hardness without any decrease in resistance to gross cracking.

Molybdenum is an effective element in improving hardenability and high temperature strength. It retards the softening of martensite at all tempering

temperatures. Above 1000°F (540°C), it partitions to the carbide phase and thus keeps the carbide particles small and numerous. In addition, molybdenum reduces susceptibility to tempering embrittlement. Chromium, like molybdenum, is a strong carbide-forming element that can be expected to retard the softening of martensite at all temperatures. Also, by substituting chromium for some of the iron in cementite, the coalescence of carbides is retarded. As a result of the stability of chromium carbide at lower austenitizing temperatures, its hardenability becomes less effective. Vanadium is a stronger carbide former than molybdenum and chromium and can therefore be expected to have a much more potent effect at equivalent alloy levels. The effect of vanadium is probably due to the formation of an alloy carbide that is very stable at elevated temperatures. The steel must be austenitized at a sufficiently high temperature and for a sufficient length of time to ensure that the vanadium or at least most of it is in solution and thus able to contribute to hardenability. Complete solution of the vanadium carbides is possible only if small amounts of vanadium are in the steel. Excess content will leave carbide particles on the grain boundaries in the quenching and tempering steel which will lower the toughness of the steel. High austenitizing temperatures can completely dissolve the carbides in H13 steel. But too high an austenitizing temperature can enhance grain growth rapidly. Silicon has been believed to precipitate on grain boundaries, martensite lath boundaries and/or martensite lath/carbide interfaces during tempering^(26,76,77). This could enhance the embrittlement of a die steel and lower its toughness. A lower silicon content in steel

K presumably minimizes the interfacial segregation and could result in higher toughness and thermal fatigue resistance^(26,76).

In view of above data, it was proposed that some of these new steels be tested for thermal fatigue resistance by using the immersion test that has been proved to be successful⁽⁴⁾ and toughness by using the Charpy V-notch impact test. The Charpy V-notch impact energy over the significant temperature range provides the transition behavior of the steels. This information is intended to measure the gross cracking resistance of the steels. These tests were conducted at hardnesses from 44 to 52 Rockwell C to cover the range of interest. The processing and testing of the new steels followed the procedures recommended for premium grade H-13 die steel. The microstructure of the steels at different heat treatment stages and condition was determined. The goal was to find the best composition and heat treatment of steels for aluminum die casting dies.

IV. TEST PROCEDURES AND EQUIPMENT

4.1. Heat Treatment

4.1.1. Annealed Structure

The steels were received in the annealed condition. In this condition, the desired microstructure is spheroidized carbides in a matrix of equiaxed ferrite grains. This is a microstructure which yields a uniform hardened structure after austenitizing and quenching. Microscopical examination of the steels in the annealed condition was conducted to identify properties-structure relationships. One of the features monitored in the study is banding caused by segregation of alloying elements during the solidification of the steels. Although some banding is anticipated, excess banding can be detrimental to the properties.

4.1.2. Optimizing the Austenitizing Temperature

Steel hardening requires austenitizing and quenching from the austenitizing temperature. The austenitizing temperature is an important variable that influences both the thermal fatigue and the fracture toughness of die steels. A higher austenitizing temperature improves the resistance to tempering which results in good

thermal fatigue resistance because of the increased solution of alloy carbides, but too high an austenitizing temperature decreases the toughness of the steel because of significant grain growth. The optimum temperature is obtained by balancing both carbides solution and grain growth.

The steels should be held at an austenitizing temperature for an appropriate period after reaching the desired furnace temperature to insure uniformity. It should also be noted that, although a long holding time provides good hardness retention, it also provides a somewhat larger grain size. Experience shows that a holding time of 30-45 minutes at the austenitizing temperature suffices for hot work tool steels⁽³⁷⁾. The austenitizing treatment at CWRU was conducted in an argon protected atmosphere tube furnace for one half hour at the temperature range from 1850°F to 2100°F, followed by oil quenching in an oil bath. The size of the test specimens was about 1/2" x 1/2" x 3/8".

Subsequent to the controlled heat treatment, the specimens were polished and the hardness of each sample was measured. Then the specimens were etched using 4% nital and studied by using Scanning Electron Microscopy (SEM) to determine the size of the grains and the solutioning of the carbides.

4.1.3. Tempering

Tempering tests were conducted for all the steels after the specimens were austenitized at 1900°F, except for Chromo-N which was austenitized at 1950°F, in an argon protected furnace for one half hour and followed by immediately oil quenching. The quenched samples were tempered for one hour at 400°F, 600°F, 800°F, 900°F, 950°F, 1000°F, 1050°F, 1100°F, 1200°F and 1300°F respectively in the same argon protected furnace. The hardness was then measured after the samples were polished and plotted as a function of the tempering temperature for each steel.

4.1.3. Cooling Rate

The Charpy V-notch impact specimens and the thermal fatigue immersion specimens, described in section 4.2 and 4.3 respectively, were produced from the same forged and rolled steel billet with identical orientation parallel to the longitudinal direction for each steel. Some of the specimens were processed by oil quenching from the austenitizing temperature. Others were heat treated in vacuum furnace and cooled at various rates by pressure controlled nitrogen gas from the austenitizing temperatures. The cooling rates were recorded by thermocouples embedded in witness blocks. Figure 4.1 shows typical cooling rate of a thermocouple embedded in a 2" x 2" x 3.8" block, one inch below the center of the 2" x 2" side. Figure 4.2 shows cooling curves measured by a thermocouple embedded inside the

wall of the thermal fatigue sample as shown in Figure 4.4. The thermocouple was located at 3 inches below the top, halfway between the corner and the bore. A summary of the cooling conditions is listed in table 4-1.

Table 4-1. Cooling rates used in the investigation

Quench Medium	Pressure (bar)	Cooling Rate (°F/min)
oil	atmosphere	>340
N ₂	10	>200
N ₂	7	>200
N ₂	5	>160
N ₂	1	<50

All specimens were processed with 0.050 inch allowance for removing any surface skin after the heat treatment where the composition could be affected by exposure to elevated temperature and the environment.

4.2. Charpy V-notch Impact Test

The Charpy V-notch impact test was used for toughness evaluation because of its widespread use, relative ease and low cost. The test specimen is the standard ASTM type A Charpy impact specimen as illustrated in Figure 4.3^(65,66). The tests were conducted in a Wiedemann-Baldwin impact machine (Model S1-1D), and the test procedures were in accordance with ASTM Specification E23-86 where applicable⁽⁶⁶⁾.

Ferritic steels and other body-centered cubic materials exhibit a transition behavior from ductile (microvoid coalescence) at high temperatures to brittle fracture (cleavage) at lower temperatures. This transition illustrates the need for designing the die operation against brittle fracture by using a service temperature above the ductile-to-brittle transition temperature for the die condition. The transition behavior of the candidate die steels was investigated by conducting the Charpy V-notch impact tests at -100°F, room temperature, 212 °F, 300 °F and 450 °F. The -100°F temperature was obtained by mixing dry ice and acetone, 212 °F by boiling water and 300 °F and 450 °F by heating silicon oil. The specimens were immersed in the various baths and kept at the desired equilibrium temperatures with the specimen tongs. The time needed to set the specimen into the test machine from the constant temperature bath before breaking was kept below 10 seconds except for the room temperature test. Otherwise the specimen and tongs were put back in the bath and re-heated or re-cooled.

4.3. Thermal Fatigue Test

4.3.1. Test Specimen and Equipment

The thermal fatigue specimen used in this evaluation of relative behavior of die steels has been employed successfully for this purpose for 25 years. The specimen is processed to the dimensions shown in Figure 4.4. It has been used to evaluate a large number of potential die materials for aluminum die casting dies⁽¹³⁾. It is 2 x 2 x 7 inch rectangular parallelepiped specimen with a 1.5 inch diameter and 6.5 inch deep hole in the center for internal water cooling. The test produces the considerable constraint and high thermal fluctuations during immersion and removal from the molten 380 aluminum alloy bath. The test equipment used for the immersion is illustrated in Figure 4.5.

The four corners of the specimen have a 0.010 inch radius, which is hand polished with 240, 320 and 400 grit silicon carbide papers, that intensifies the predominately uniaxial stress at this location. The outer surface of the specimen is sprayed with a commercial water-base die lubricant (Chem-Trend, Safety-Lube 8700) diluted 50 to 1 with water just before it enters the molten aluminum bath. The optimum level of the lubrication was carefully adjusted and the test specimen was turned 90° along the long axis of the specimen every 1,500 cycles to insure the uniform application of the lubricants. By the decreasing the temperature of the die

surface through the evaporation of the corner liquid as shown in Figure 4.6⁽¹⁸⁾, lubricants affect the formation of thermal stresses which lead to the heat checking. Water flows through the specimen at a rate of 1.5 gallon per minute. The internal water cooling system is shown in Figure 4.7. The molten bath is maintained at 1350°F and the specimen is immersed for 12 seconds and then removed from the bath for 24 seconds to produce thermal fluctuations. Previous studies^(8,13) have showed that a corner of 0.010 inch radius undergoes a temperature fluctuation of 200°F-1175°F at the corner of the specimen with a 12 second heating and 24 second cooling cycle. The resulting typical thermal cycle at the corner surface of the specimen is shown in Figure 4.8⁽¹³⁾. The thermal effects or the differences in external and internal steel temperature obtained in this test are more severe than the conditions encountered in normal die casting operations^(13,22).

4.3.2. The Evaluation of Thermal Fatigue Resistance

The standard procedure is to operate the test for 5,000 immersion cycles, measure the cracking pattern and follow this method for 10,000 and 15,000 total cycles. After each 5,000 cycles, the specimen was removed from the testing unit and the fatigue crack initiation and growth were evaluated. The oxide layers that had formed during cycling test were removed by manual polishing with 240, 320, 400 silicon carbide papers along the length of the corner. Since the maximum temperature

that is attained is at the corner or edge of the immersion specimen and the stress state is uniaxial at the edge, thermal fatigue cracks initiate at the corner and then propagate internally. Therefore, special care was needed for corner preparation. The papers were clamped in a V-notched block to achieve the required radius. The radius was checked with a 0.010 inch radius gauge on each corner. A 3 inches long section on the corners equidistant from each end was used to measure the cracks at 100X and recorded in 100 micron ranges for depth. Only the center 3 inches of each corner was measured to eliminate possible end effects⁽¹³⁾. The crack measurements were obtained at a magnification of 100x using a Leitz microhardness tester.

The cracking pattern is characterized as two parameters, namely the average maximum crack length L_a and the total crack area A_t to evaluate the thermal fatigue behavior of the specimens. The average maximum crack length is the average length of the longest crack of each of the four corners within the middle three inches of the corners:

$$L_a = \frac{1}{4} \sum_{i=1}^4 L_{mi} \quad (4-1)$$

where $i = 1, 2, 3, 4$ indicates each of the four corners,

L_{mi} = maximum crack length of # i corner.

The total crack area is the sum of the product of the number of cracks in each 100 micron size range and the square of the midpoint of that range for all the four corners within the middle three inches of the corners:

$$A_t = \sum_{i=1}^4 \sum_{j=1}^n N_{i,j} L_j^2 \quad (4-2)$$

where $L_j = 100j - 50 \mu\text{m}$;

$j = 1, 2, \dots, n$, corresponding different crack length range;

$N_{i,j}$ = number of the cracks of # i corner in the crack length range of $100(j-1)$ to $100j \mu\text{m}$.

The more severe the crack pattern, the bigger value of the L_a and A_t and the lower the thermal fatigue resistance of the tested material. The results of this test have correlated closely with the behavior of dies in industry⁽⁴⁾.

4.4. Electro-Discharge Machining

Based on previous work^(64,67), electro-discharge machining (EDM) affects the steel surface and can reduce the thermal fatigue resistance below that for surfaces that have been conventionally machined. The detrimental influence of EDM is attributed to the damaged layers consisting of a brittle recast layer, a hardened layer and a softer

over-tempered region, as well as the tensile residual stresses introduced by the processing. The damage is especially severe when a high energy, rapid removing step processing is applied. A commercial EDM process that uses multiple small steps of decreasing energy followed by a grit polishing to remove the brittle recast layer and tempering at 50°F below the maximum temperature used to soften the hardened layer minimizes the effect of EDM on thermal fatigue cracking behavior.

All thermal fatigue specimens used for the EDM evaluation were austenitized at 1875°F and quenched in oil to obtain the optimum microstructure. Commercial EDM procedures using multiple small steps of decreasing energy to remove the final 0.050 inch of material from the surfaces were employed. To simulate the behavior of real dies, the procedures followed in the preparation of these samples closely follow the die finishing operation. Only three of the four corners of each EDM specimen were processed by EDM for the evaluation, the fourth one was finished using the normal grinding operations as a control or reference.

Figure 4.1. Cooling Curves for Impact and Thermal Fatigue Samples

Figure 4.2. Cooling Curves for Thermal Fatigue Samples

Figure 4.3. ASTM Standard Type A Charpy V-Notch Specimen and the Striker-Anvil Arrangement

Figure 4.4. Thermal Fatigue Specimen

Figure 4.5. Thermal Fatigue Test Equipment

Figure 4.6. An Idealized Temperature Cycle During the Die Casting Process

Figure 4.7. The Internal Water Cooling of the Thermal Fatigue Specimen

Figure 4.8. Temperature Profile at Specimen Corner Surface

V. RESULTS AND DISCUSSIONS

5.1. Heat Treatment

5.1.1. Annealed Structure

The steels were received as-annealed. In this condition, the carbides are spheroidal as depicted in Figure 5.1 (a)-(d) for the different steels. K steel has a more uniform carbide distribution without carbide stringers. This is attributed to the less contents of alloying elements in this steel. Minor differences in banding present in the steels has also been determined as shown in Figure 5.2 (a)-(d).

5.1.2. Optimizing the Austenitizing Temperature

The austenitizing was carried out at 1850°F, 1900°F, 1950°F, 2000°F and 2100°F as described in the previous chapter. The resulting microstructures for each one of the austenitizing temperatures are shown for each steel in Figure 5.3. The most obvious feature in the micrographs are the carbides. After austenitizing at 1850°F, H-13 still shows a lot of undissolved carbides while K only has a few left. C and Q are in-between. After austenitizing at 1900°F, H-13 still has a number of undissolved carbides while K has very few left. Austenitizing at 1950°F or above leaves

practically no carbides in K. Austenitizing at 2000°F eliminates the carbides from all the steels. This difference in the behavior of the steels can be attributed the lower alloying content of K, in particular the carbide forming element Vanadium.

The dependency of the grain size on the austenitizing temperature is shown in Figure 5.4 and the hardness dependency is shown in Figure 5.5. P.G. H-13 has a consistently smaller grain size than K over the entire austenitizing temperature range. This is attributed to the larger carbide contents that inhibit grain growth. The grain size of K increases sharply between 1950°F and 2000°F. A parallel, but less drastic grain growth occurs in H-13 over a broader range of austenitizing temperatures, also starting around 1950°F. The hardness of H-13 is slightly lower (57.5-60Rc) than K in the 1850°F to 1950°F austenitizing temperature range, and becomes higher (60-61Rc) in the 1950°F to 2100°F range. These results are consistent with the microscopic observation: while in the K carbides are completely dissolved at 1950°F, in the H-13 it takes a higher austenitizing temperature to entirely bring them into solid state solution.

The hardness at elevated temperature of the steels was measured in this condition and is shown in Figure 5.6.

The grain sizes of Q and C are similar and between that of K and H-13 over a wide austenitizing range as shown in Figure 5.4. The smaller grain sizes at 2100°F are attributed to the higher overall alloy element content in Q and C. The grain sizes

increase dramatically between 1950°F and 2000°F. The hardness of Q and C is lower than K and H-13 over the entire 1850°F to 2100°F austenitizing temperature range. These results are consistent with the lower carbon content which also affects its hardenability. Based on the above results the austenitizing temperatures selected for further testing of thermal fatigue and Charpy V-notch impact specimens are 1875°F to 1925°F.

5.1.3. Tempering

The hardness as a function of the tempering temperature for each steel is shown in Figure 5.7(a) and (b). Figure 5.7(a) shows the tempering curve in the entire temperature range from room temperature (as quenched) to 1300°F. Figure 5.7(b) shows the more significant details for the tempering temperature from 900°F to 1300°F.

The steel in as-quenched condition is hard but it is also very brittle. Tempering is the heat treatment of hardened steels to reduce the brittleness and increase the toughness as its major objective. Softening also occurs during tempering which reduces the strength of a steel. Alloying elements in carbon steels help to retard the rate of softening during tempering. Steels for aluminum die casting dies experience a high temperature that could reach 1200°F during the casting thermal cycling. Softening resistance is one of the major concerns in die steel selection.

Figure 5.7 shows that K steel has a similar softening resistance to H-13 in the higher die tempering temperature range from 1050°F to 1150°F. Q has the highest softening resistance, and C has the lowest softening resistance. Tempering C for one hour at 1100°F results in a drop from as-quenched hardness 55-56 Rc to 42 Rc. This hardness is lower than desirable for aluminum die casting dies. The optimized tempering should include 1 hour at 1040°F followed by a second 1-2 hours tempering at 1000°F to reach a 46 Rc hardness level.

As discussed in Chapter II, the die surface temperature for aluminum casting can reach to 1100°F or even higher in squeeze casting. Therefore, C steel would be expected to exhibit a low resistance to thermal fatigue in aluminum die casting die applications. The thermal fatigue tests confirmed this conclusion.

5.2. Charpy V-notch Impact Properties

All the steels in the investigation were subjected to the Charpy V-notch impact test to evaluate their toughness. The brittle-ductile transition behavior was also determined with the test conducted at -100°F, 72°F (room temperature), 220°F, 300°F and 450°F. The results for each steel are shown in Figures 5.8 to 5.11. Table 5-1 indicates the Charpy V-notch impact energy at room temperature (72°F) for different quenching rates.

Table 5-1. Charpy V-notch impact energy at room temperature (ft.lb)

QUENCH RATE (°F/min)	STEEL			
	P.G. H-13	K	Q	C
oil (> 340)	14.5 - 16.0	16.8 - 17.5	8.0	6.5
7-10 bar N ₂ (> 200)	12.2 - 14.5	14.5 - 16.2	4.0	4.5 - 6.5
5 bar N ₂ (>160)	14.0	14.0		
1 bar N ₂ (< 50)	8.0	11.5		

It has been reported that the room temperature Charpy V-notch impact energy values exceeding 11 ft.lb (15 Joule) are more desired for aluminum die casting die applications⁽⁶⁸⁾. Using this room temperature guideline, K is the only steel tested with acceptable values at all of the quenching rates tested. Premium grade H-13 is acceptable except for the lowest cooling rate (1 bar). Q and C are completely unacceptable. It is pointed out that complete transition curves are more significant in measuring the toughness behavior of die steels. Steels with low toughness may be subject to gross cracking in die thermal cycling. In this investigation, two thermal fatigue samples for Q and C steels with low Charpy V-notch impact energies failed by gross-cracking during the thermal cycling test as indicated in Table 5-2.

The data in Figures 5.8 to 5.11 confirms the significant increase in the fracture toughness or impact resistance at higher temperature for all of these steels. It also indicates higher toughness for the steels that were quenched in oil, or the fastest cooling rates. The toughness, in general, is inversely proportional to the hardness in the range of interests under the same cooling condition. The effects of the cooling rate and hardness will be discussed in detail in section 5.5 and 5.6 respectively.

5.3. Thermal Fatigue Properties

Table 5-2 lists the thermal fatigue specimens tested with the heat treatment information for the four steels. H-13 steel specimens were numbered with the beginning of upper case letter H, K steel with letter K, Q steel with letter Q and C steel with C; then followed by an Arabic number. The EDMed specimens are indicated in the last column in the table. Only three of the four corners were EDMed in the EDMed samples and the another one was ground as a build-in reference. In this way, the specimen serves two purposes. The results are identified with the corresponding sample identification number followed by the lower case e for EDMed and g for ground respectively when applicable. The results of the total crack area

Table 5-2. List of thermal fatigue specimens

#	Steel	T _a (°F)	Quench	1st temp	2nd temp	HRc	Other
H1	H-13	1875	Oil	1050°F/2h	1100°F/2h	48	
H2	H-13	1925	Oil	1050°F/2h	1100°F/2h	47	
H3	H-13	1875	Oil	1100°F/2h		45	
H4	H-13	1875	5bar, N ₂	1050°F/2h	1100°F/2h	50	
H5	H-13	1875	1bar, N ₂	1100°F/1h	1100°F/1h	52	
H8	H-13	1875	Oil	1100°F/2h	1100°F/1h	48	EDMed
H10	H-13	1875	Oil	1100°F/1h	1100°F/1h	50	EDMed
K1	K	1875	Oil	1050°F/2h	1100°F/2h	46	
K2	K	1925	Oil	1050°F/2h	1100°F/2h	45	
K3	K	1875	7bar, N ₂	1050°F/2h	1100°F/2h	49	
K4	K	1875	Oil	1100°F/2h	1100°F/1h	44	
K5	K	1875	1bar, N ₂	1100°F/1h	1100°F/1h	50	
K8	K	1875	Oil	1100°F/2h	1050°F/1h	46	EDMed
K10	K	1875	Oil	1100°F/1h	1100°F/1h	49	EDMed
Q1	Q	1875	10bar, N ₂	1100°F/1h	1130°F/1h	51	
Q2	Q	1875	Oil	1100°F/1h	1130°F/1h	50	
Q3	Q	1875	1bar, N ₂	1100°F/1h	1130°F/1h	50	
C1	C	1875	10bar, N ₂	1000°F/1h	1030°F/1h	46.5	
C2	C	1875	Oil	1000°F/1h	1030°F/1h	47	
C3	C	1875	1bar, N ₂	1000°F/1h	1030°F/1h	49	

Table 5-3. Hardness change (softening) with thermal cycling (Rc)

#	Steel	Thermal Cycling				
		0	5,000	10,000	15,000	20,000
H1	H-13	48	47	46.5	46	
H2	H-13	47	46.5	46.5	46	46
H3	H-13	45	45	Failed by dropping in Al bath		
H4	H-13	50	47	46.5	46	
H5	H-13	52	50	48	47	
H8	H-13	48	47.5	47.5	47	47
H10	H-13	50	50	49	49	
K1	K	46	45.5	45	45	45
K2	K	45	45	45	45	45
K3	K	49	47.5	47	47	47
K4	K	44	43.5	43	43	
K5	K	50	50	50	48	
K8	K	46	46	46	45.5	45.5
K10	K	49	48.5	48	48	
Q1	Q	51	50.5	50.5	50	50
Q2	Q	50	48	failed		
Q3	Q	50	50	50	failed	
C1	C	46.5	40	37	failed	
C2	C	46	39	36	34	
C3	C	49	40	35	failed	

were normalized as a four corner sample for both EDMed and ground corners, although only three and one corners for EDMed and ground respectively are present.

The results of the thermal cycling testing are shown: in Figures 5.12(a) and 5.12(b) for H-13; in Figures 5.13(a) and 5.13(b) for K; in Figures 5.14(a) and 5.14(b) for Q; and in Figures 5.15(a) and 5.15(b) for C. The results are represented as the Average Maximum Crack Length and the Total Crack Area for each steel. The heat treating conditions and as-tempered and tested hardness are labeled for each curve. Notice that higher values, or the upper curves, indicate longer cracks or a larger area of cracks. The higher a curve is on the plot, the worse its relative thermal fatigue resistance. The tempered hardness of the evaluated samples was in the range of 44-52Rc. The hardness change (softening) with the thermal cycling is given in Table 5-3.

Figures 5.16 and 5.17 show examples of the total crack number distributions of the four corners for thermal fatigue samples H4 and H5 developed after 5,000, 10,000 and 15,000 cycles in terms of crack length. All the specimens had the similar distribution pattern. Figure 5.18(a) and (b) are micro-photos taken on the surface of the center area of one corner of the specimen H4 after 15,000 cycles. The (a) shows a few cracks developed on the center area of the corner at 50X. The (b) shows the detail of one single crack on this location at 400X and also shows clearly that the crack was wedged by oxides.

It is noticed that Figures 5.16 and 5.17 have the same scale of crack length and a considerably different (more than 3 times) scale of total crack numbers. Which means that H4 and H5 samples have the same crack length range in Figure 5.12(a), but H5 has a higher number of cracks than H4 does. The total crack area is defined as proportional to the general crack pattern, so the values of the total crack area for H4 and H5 are well separated in Figure 5.12(b). The total crack area is very sensitive to the crack length because it is proportional to the square of the crack length. Generally, a sample with many small cracks would have a lower crack area than a sample which contained fewer deep cracks. Of course, the total crack area value may equilibrate if a sufficient number of small cracks exists compared to one larger crack. For instance, one single 500 μm crack is equivalent to one hundred 50 μm cracks in terms of the total crack area; if the longer one is 5000 μm deep, the equivalent number of 50 μm smaller ones would be 10,000. Nevertheless, the crack area is a good indicator of heat checking properties and has been supported by other investigations as well^(33,69-72).

The results in Figures 5.12 and 5.13 clearly demonstrated, by comparing the scale ranges of the total crack area axes in Figures 5.12(b) and 5.13(b) or $20 \times 10^6 \mu\text{m}^2$ for H-13 compared to $4 \times 10^6 \mu\text{m}^2$ for K, the ratio is 5. That the overall thermal fatigue resistance of K is significantly superior to premium grade H-13. The oil quenched, 1925°F austenitized K sample K2 did not develop any thermal fatigue cracks after the standard 15,000 cycles. Testing was extended to 20,000 cycles. This extension still did not generate any thermal fatigue cracks on the four corners of the

thermal fatigue sample. For oil quenched, 1875°F austenitized specimen K1 and K10g only a few fine cracks were formed after 10,000 cycles. This also demonstrated that higher austenitizing temperature improves thermal fatigue resistance because of more alloying elements in solution at higher austenitizing temperatures which enhance the resistance to tempering and heat checking. The cooling rate obviously has a marked effect on the thermal fatigue results as will be discussed in the section 5.5.

Only three thermal fatigue specimens were tested for each of Q and C steels because of the catastrophic gross cracking failure that occurred for both of the steels. The Q samples show good resistance to thermal cycling as demonstrated by the curves in Figure 5.14(a) and 5.14(b). However, gross cracking was encountered with the steel as indicated in Table 5-2. Figure 5-19 shows one half of a gross cracked Q sample Q3. This specimen was split to two parts starting from the bottom of the sample. This catastrophic failure might be attributed to the higher alloy content of this steel. The purpose of the higher alloying elements is to improve the softening resistance and heat checking resistance. The resistance of this steel to tempering was confirmed, as shown by the tempering curve in Figure 5.7. But the excess alloy content produces large carbide particles on the grain boundaries in the quenched and tempered steel which enhance the brittleness of the steel and results in the lower toughness and possible gross cracking. The measured Charpy V-notch impact energies are only in the range 4 to 8 ft.lb at room temperature for some of the heat treated samples. High austenitizing temperature can completely dissolve the carbides in solution. However,

too high austenitizing temperature will enhance grain growth rapidly and result in the same detrimental results.

The thermal fatigue test data of the C samples shown in Figure 5-15(a) and 5-15(b) is fairly close to H-13 for the early cycling, although the data is not complete because of early gross-cracking failure. However, these specimens spalled locally along the corners, particularly at the hottest or bottom end of the specimens. Two of the specimens C1 and C3 were catastrophically gross cracked when running the third of the 5,000 cycles. One specimen was split by a gross crack from the bottom of the specimen and another one from the top as shown in Figure 5-20. It is also noted from Table 5-3 that the hardness of the thermal cycling samples dropped dramatically from 46-49 Rc as heat-treated to the middle 30's during the thermal cycles. This is consistent with the results of the tempering tests discussed previously. One of the causes for this lower hardness is the lower carbon content (0.26%). It was also noticed that C has lowest toughness over a wide range of temperature among these tested steels as shown in Figure 5.11. The softening, low toughness and gross cracking indicate that C steel is not as suitable as the other steels for aluminum die casting die applications.

Based on the results of both toughness and thermal fatigue tests. At this point, it was concluded that the performance of both Q and C steels was relatively poor for aluminum die casting dies. The objective of this study was to identify the better steel available on the market and the best processing of these steels for aluminum die

casting dies. For these reasons, the work has been mainly concentrated on premium grade H-13 and K steels.

5.4. Effect of Austenitizing Temperature on Toughness and Thermal Fatigue Resistance

Two austenitizing temperatures 1875°F and 1925°F were selected for the oil quenched H-13 and K steel Charpy V-notch impact and thermal fatigue samples to investigate the austenitizing temperature effect on toughness and thermal fatigue resistance. The results indicate that the Charpy V-notch impact energies for the samples austenitized at 1875°F are higher than that for the samples austenitized at 1925 °F at the same hardness level as shown in Figures 5.8 and 5.9 for H-13 and K respectively. The higher austenitizing temperature dissolves more alloying elements in steel matrix and strains the matrix after quenching more than at lower austenitizing temperatures. The more strain the matrix has, the less ductile the matrix is, hence, the lower toughness will be expected.

However, Figures 5.13 and 5.14 show that the higher austenitizing temperature produces higher thermal fatigue resistance for both H-13 and K steels. The 1925°F austenitized K sample K2 did not develop any thermal fatigue cracks after the standard 15,000 cycles. Testing was extended to 20,000 cycles, but still did not generate any thermal fatigue cracks on the four corners of the thermal fatigue

sample. Both the average maximum crack length and total crack area curves for the 1925°F austenitized K steel sample K2 lie on the zero value for the both of the parameters over the entire thermal cycles as shown in Figures 5.13(a) and 5.13(b). The curves for the corresponding 1925°F austenitized H-13 steel sample H2 in Figures 5.13(a) and 5.13(b) are also provide the lowest values for total crack area. The higher austenitizing temperature improves thermal fatigue resistance because of more alloying elements in solution at higher temperature which resist tempering increase the thermal fatigue resistance of these steels.

5.5. Effect of Cooling Rate on Toughness and Thermal Fatigue Resistance

The cooling rates for quenching the Charpy V-notch impact and thermal fatigue specimens from the austenitizing temperature carried out in this investigation are shown in Table 4-1, Figures 4.1 and 4.2 as discussed in section 4.1.4.

Figures 5.21 and 5.22 provide the effect of cooling rate on the Charpy V-notch impact energy for H-13 and K for all tested temperatures. While Table 5-1 shows the effect of cooling rate for H-13, K, Q and C at room temperature. It has been well demonstrated that the toughness increases with increasing cooling rate at all temperatures. This improvement in Charpy V-notch impact results is much more marked at higher temperatures. At the lowest cooling rate, one bar N₂ quenching to

provide the cooling rate of less than 50 °F/min, K has higher toughness than H-13 over the entire temperature range.

The same behavior applies to the effect of cooling rate on the thermal fatigue resistance which is shown in Figures 5.23(a)-(b) and 5.24(a)-(b) for H-13 and K respectively. Figure 5.23(a) and Figure 5.24(a) provide the effect of cooling rate on the average maximum crack length and Figure 5.23(b) and Figure 5.24(b) show the effect on the total crack area. The cooling rate has a marked effect; the thermal fatigue resistance improves greatly with faster cooling from the austenitizing temperature..

The effect of cooling rate on the toughness and thermal fatigue resistance can be rationalized by the continuous cooling transformation (CCT) diagram. Figure 5.25 shows the CCT diagram of H-13 steel austenitized at 1900°F and the effects of the cooling rate on the microstructure and toughness⁽⁷³⁾. Carbides precipitation on grain boundaries is one cause of reduced toughness and thermal fatigue resistance at slow cooling rates when the austenite transforms into bainite or even pearlite, it also can lower the properties of the die steels significantly. The cooling rate in the temperature range 1750°F to 550°F should be at least 55°F/min to avoid excessive intergranular carbide precipitation and to obtain the desirable structure and properties for H-13 steel, as shown in Figure 2.2^(49,50). The cooling rate in a one bar N₂ vacuum furnace employed in this investigation is less than 50°F/min. This slow cooling provides a bainite structure with excessive grain boundary carbides.

Altering the alloy composition from the H-13 steel analysis has been shown to improve the properties of die steels based on the thermal fatigue and toughness tests conducted. One of these steels is K that has a lower silicon and vanadium contents than H-13. These changes have reduced the amount of carbides that are present and the higher molybdenum content of this steel may also have played a role in the better properties. Vanadium is a strong carbide forming element and requires a high austenitizing for a sufficient length of time to obtain the solution of these carbides into the austenitic matrix. Any undissolved carbides will act as potential crack starters and may be sites for further carbide precipitation that occurs as the grain-boundary carbides area shown in the CCT diagram in Figure 5.25. Silicon is also considered to promote the grain boundary segregation. Lower vanadium and silicon contents move the nose of the grain-boundary carbide curve to the right or longer times in the CCT diagram of the steel. This change makes K less susceptible to damage at slower cooling rates than H-13 to the cooling rate as indicated by the data obtained in this thesis. K is higher in molybdenum and this increases the strength of the matrix at high temperatures. Molybdenum is also a strong carbide former but dissolves into the austenite solution at lower temperatures than vanadium.

5.6. Effect of Hardness on Toughness and Thermal Fatigue Resistance

The effect of hardness on toughness has been very well studied^(8,34,37,42,47,50) and also demonstrated in this investigation. Under the same quenching condition from austenitizing temperatures the toughness of die steels increases with increasing ductility and is inversely proportional to the hardness in the range of interest. Higher hardness improves the thermal fatigue resistance because of the higher strength of harder steels. The degree to where the hardness level of a die can be increased is limited by the loss in toughness that decreases at the higher hardness. H-13 steel with Rc 46-47 hardnesses is usually employed because this hardness level has the best combination of good thermal fatigue resistance and toughness for aluminum die casting dies^(50,64).

Figures 5.26(a)-(b) and 5.27(a)-(b) show the effect of hardness on the thermal fatigue resistance for H-13 and K steels respectively. All samples for the data in these figures were austenitized at 1875°F, oil quenched, and double or triple tempered to the final hardness. Figure 5.26(a) and Figure 5.27(a) show the effect of hardness on the average maximum crack length, and Figure 5.26(b) and Figure 5.27(b) demonstrate the effect on the total crack area. It is shown that the thermal fatigue resistance for both steels is improved by increasing the hardness up to 49/50 Rc. From Figures 5.27(a) and (b) it is clearly shown that the thermal fatigue resistance for K steel increases sharply when the hardness increases from Rc 44 to 46. The rate of this increase slows down with further increases in the hardness. This indicates a minimum of Rc 46 hardness is preferred for K steel for aluminum die casting dies. However, the

toughness has to be considered due to increasing susceptibility to gross cracking at higher hardness especially at lower cooling rates from the austenitizing temperature. It appears that a hardness of Rc 46 up to 50 Rc may be used for dies quenched at high cooling rate such as oil quenching to take advantage of the high thermal fatigue resistance. Lower hardnesses such as 46/47 Rc is probably preferred for dies cooled at a lower rate such as one bar N₂ gas quenching in a vacuum furnace. Large dies are more susceptible to gross cracking and may be best employed at lower hardness levels.

5.7. Effect of EDM on Thermal Fatigue Properties

Four EDMed thermal fatigue specimens H8, H10, K8 and K10 were tested as listed in the Table 5-2. Two were premium grade H-13 steel and the other two were K steel as identified by H and K. All the specimens were austenitized at 1875°F and quenched in oil to obtain the optimum microstructure, and then double tempered to their final hardnesses. Three of the four corners for the specimens were EDMed to remove the final 0.050 inch of material from the surfaces in multiple small steps of decreasing energy as in the commercial die finishing operation and the fourth corners in each one were ground and polished by the standard method used in the investigation. The H8 and K8 specimens were also post-EDM treated by tempering at 1050°F to soften the hardened layer and grit polished to remove the brittle recast

layer. However, the H10 and K10 specimens were tested in the as-EDMed condition without removal of the white layer. The EDMed corners on H8 and K8 were EDMed to sharp corners and then hand-polished to the 0.010" radius used for all thermal fatigue specimens. This polishing of the EDMed surface removed more material at the corners than the nearby flat surface and further reduced the EDM effect. The EDMed corners on the no post-EDM treated H10 and K10 specimens were directly EDMed to 0.010" radius by using a specially shaped electrode without any polishing.

Figure 5.28(a) shows the cross sectional view of the EDMed specimen K8. No any recast layer remains on the surface and the surface of this specimen is smooth because of polishing after EDM. The recast layer however is present on the surface of the no post-EDM treated specimen K10 as demonstrated in Figure 5.28(b). This layer was formed by steel melted by the spark during EDM and then resolidified when the treatment was complete. The recast layer appears as a white non-etchable layer and is approximately 5 to 10 μm . This layer is uneven and contains resolidified droplets on the surface. There is also a hard brittle untempered martensite layer immediately under the white layer which formed below the surface that reached temperatures within the austenitic range of the steel and then rapidly cooled. This untempered layer is not clearly detected because it is so thin due to the multiple small steps of decreasing energy EDM. Tempering occurs in the layers further into the material that were heated only into the tempering range of the steel. This untempered layer is about 50 μm thick for both samples K10 and K8 as shown in Figure 5.28. The existence of

these layers is demonstrated by measuring the microhardness of the cross sections of these samples. Figures 5.29(a) and (b) show the indentation of Vickers hardness on the cross sections of EDMed specimens K8 and K10 respectively. The resulting microhardness along the cross sections are shown in Figure 30 for these two samples. The microhardness as measured on the Vickers scale within 10 μm is not included because satisfactory readings could not be obtained. The over-tempered layers are present in both samples as clearly demonstrated in Figure 30.

Little differences occurred in thermal fatigue cracking on EDMed and ground corners for post-EDM treated samples H8 and K8. The overall thermal fatigue results are shown in Figures 5.12(a)-(b) and Figures 5.13(a)-(b) labeled as H8 and K8 respectively without distinguishing the EDMed and ground corners. The results are nearly identical to those obtained for the other ground samples similarly heat treated for each steel. The post-EDM treatment of tempering the hardened layer and removing the brittle recast layer of the specimen after EDM process avoided any loss of thermal fatigue resistance..

The normalized results for the specimens without post-EDM treatment H10 and K10 are shown in the same Figures 5.12(a)-(b) and Figures 5.13(a)-(b) labeled as H10g, H10e, K10g and K10e. The g indicates ground corners and e indicates EDMed corners. The EDMed corners had considerably higher average crack length and total crack area. The brittle recast and untempered hardened layers were contributed to the reduced thermal fatigue resistance for these specimens. These brittle layers enhanced

the initiation of cracks during the thermal cycling. Even so, the results were still better than those of vacuum heat treated and one bar N_2 gas quenched specimens for both H-13 and K steels respectively.

VI. CONCLUSIONS

1). The K steel has demonstrated a higher thermal fatigue resistance than premium grade H-13 steel. K is also less susceptible to a loss in the thermal fatigue resistance at slower cooling rates from the austenitizing temperature. This superior performance is attributed to the lower silicon and vanadium and higher molybdenum present in this steel. A higher thermal fatigue resistance as measured by the thermal fatigue test should provide a longer die life. Q and C steels are poor in performance due to their low toughness and also the premature softening of C. The lower toughness makes these steels more susceptible to gross cracking.

2). Faster cooling rates from the austenitizing temperature provide both higher thermal fatigue resistance and toughness. Oil quenching provides the fastest cooling rates of higher than 340 °F/min and the best thermal fatigue resistance and toughness. Seven to ten bar N₂ gas quenching in a vacuum furnace results in the cooling rates of about 200 °F/min and a good thermal fatigue resistance and toughness. One bar N₂ gas quenching provides the cooling rates of less than 50 °F/min and a poor thermal fatigue resistance and toughness. Five bar quenching provides the cooling rates of about 160 °F/min and results in a fair thermal fatigue resistance and toughness between those for seven to ten bar and one bar cooling rates.

3). Higher austenitizing temperatures such as 1925°F compared to 1875°F provide better thermal fatigue resistance. However, lower austenitizing temperatures of 1875°F provide better toughness.

4). Higher hardness improves thermal fatigue resistance, but reduces toughness. A minimum of Rc 46 hardness is desired for aluminum die casting dies except for the very large dies because of gross cracking problems. Higher hardness can be tolerated up to 50 Rc at faster cooling rates such as oil quenching without gross cracking for smaller dies. Lower hardness should be employed for slower cooling rates because of toughness or gross cracking considerations.

5). Electro-Discharge Machining can reduce the thermal fatigue resistance compared to conventional machining operations. When the optimum EDM process of multiple small steps of decreasing energy and post-EDM treatments that remove the white layer and soften the untempered martensitic layer are employed, the effect can be reduced to a very slight amount.

(a) P.G. H-13, 800X. 4% nital etched

20 μ m

(b) K, 800X. 4% nital etched

20 μ m

Figure 5.1. Optical Micrographs of Annealed Structure

(c) Q, 800X.

4% nital etched

20 μm

(d) C, 800X.

Vilella's reagent etched

20 μm

Figure 5.1. Optical Micrographs of Annealed Structure

(a) P.G. H-13, 50X. 4% nital etched

500 μm

(b) K, 50X. 4% nital etched

500 μm

Figure 5.2. Optical Micrographs of Banding

(c) Q, 50X.

4% nital etched

500 μ m

(d) C, 50X.

Vilella's reagent etched

500 μ m

Figure 5.2. Optical Micrographs of Banding

(a-1) $T_a = 1850^\circ\text{F}$, P.G. H-13

(a-2) $T_a = 1850^\circ\text{F}$, K

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(a-3) $T_a = 1850^\circ\text{F}$, Q

(a-4) $T_a = 1850^\circ\text{F}$, C

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(b-1) $T_a = 1900^\circ\text{F}$, P.G. H-13

(b-2) $T_a = 1900^\circ\text{F}$, K

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(b-3) $T_a = 1900^\circ\text{F}$, Q

(b-4) $T_a = 1900^\circ\text{F}$, C

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(c-1) $T_a = 1950^\circ\text{F}$, P.G. H-13

(c-2) $T_a = 1950^\circ\text{F}$, K

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(c-3) $T_a = 1950^\circ\text{F}$, Q

(c-4) $T_a = 1950^\circ\text{F}$, C

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(d-1) $T_a = 2000^\circ\text{F}$, P.G. H-13

(d-2) $T_a = 2000^\circ\text{F}$, K

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(d-3) $T_a = 2000^\circ\text{F}$, Q

(d-4) $T_a = 2000^\circ\text{F}$, C

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(e-1) $T_a = 2100^\circ\text{F}$, P.G. H-13

(e-2) $T_a = 2100^\circ\text{F}$, K

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

(e-3) $T_a = 2100^\circ\text{F}$, Q

(e-4) $T_a = 2100^\circ\text{F}$, C

Figure 5.3. Effect of Austenitizing Temperature on Microstructure

Figure 5.4. Grain Size vs. Austenitizing Temperature

Figure 5.5. Hardness after Quench

Figure 5.6. Hardness at Elevated Temperature

Figure 5.7(a). Hardness vs. Tempering Temperature

Figure 5.7(b). Hardness vs. Tempering Temperature

Figure 5.8. Charpy Impact Resistance of H-13

Figure 5.9. Charpy Impact Resistance of K Steel

Figure 5.10. Charpy Impact Resistance of Q Steel

Figure 5.11. Charpy Impact Resistance of C Steel

Figure 5.12(a). Average Maximum Crack Length of H-13

Figure 5.12(b). Total Crack Area of H-13

Figure 5.13(a). Average Maximum Crack Length of K Steel

Figure 5.13(b). Total Crack Area of K Steel

Figure 5.14(a). Average Maximum Crack Length of Q Steel

Figure 5.14(b). Total Crack Area of Q Steel

Figure 5.15(a). Average Maximum Crack Length of C Steel

Figure 5.15(b). Total Crack Area of C Steel

Figure 5.16. Crack Distribution of H-13 Sample H4

Figure 5.17. Crack Distribution of H-13 Sample H5

(a) 50X

500 μm

(b) 400X

50 μm

Figure 5.18. Cracks on H-13 Sample H4 after 15,000 Cycles
(1875°F/5barN₂/Rc50)

Figure 5.19. Gross Cracked Q Thermal Fatigue Sample Q3
(1875°F/1barN₂/Rc50)

Figure 5.20. Gross Cracked C Thermal Fatigue Sample C3
(1875°F/1barN₂/Rc49)

Figure 5.21. Effect of Cooling Rate on Toughness of H-13

Figure 5.22. Effect of Cooling Rate on Toughness of K Steel

Figure 5.23(a). Effect of Cooling Rate on Crack Length of H-13

Figure 5.23(b). Effect of Cooling Rate on Crack Area of H-13

Figure 5.24(a). Effect of Cooling Rate on Crack Length of K Steel

Figure 5.24(b). Effect of Cooling Rate on Crack Area of K Steel

(a). CCT Diagram of H-13 Austenitized at 1900°F

Fast cool	A	Tempered martensite—no carbides	Tough
	C	Lower bainite—no carbides	
	B	Tempered martensite + grain boundary carbides	
	D	Tempered martensite + bainite + grain boundary carbides	
Slow cool	E	Upper bainite + large carbides	Brittle

(b). Variation in Toughness as a Function of Carbides Ejected During Cooling, Hardness Maintained at Constant Value

Figure 5.25. Effect of Cooling Rate on Microstructure and Toughness of H-13

Figure 5.26(a). Effect of Hardness on Crack Length of H-13

Figure 5.26(b). Effect of Hardness on Crack Area of H-13

Figure 5.27(a). Effect of Hardness on Crack Length of K Steel

Figure 5.27(b). Effect of Hardness on Crack Area of K Steel

(a). EDMed Thermal Fatigue Sample K8 Followed
by Tempering and Polishing. 4% Nital etched

20 μm

800X

(b). EDMed Thermal Fatigue Sample K10 without
Post-EDM Treatment. 4% Nital etched

20 μm

800X

Figure 5.28. Cross Sectional View of EDMed Samples

(a). EDMed Thermal Fatigue Sample K8 Followed by Tempering and Polishing

(b). EDMed Thermal Fatigue Sample K10 without Post-EDM Treatment

Figure 5.29. Indentation of Vickers Hardness
on the Cross Sections of EDMed Samples

Figure 5.30. Microhardness along the Cross Sections of EDMed Thermal Fatigue Samples of K Steel

VII. IN-PLANT EVALUATION

A die insert initially selected for the testing is an Ejector Center Insert. It is used in making a cover for an automotive transmission. The die has four identical die cavities with these inserts. The inserts are notorious for their short life time. Based on our recommendations, inserts were made of K and Premium Grade H-13. We also recommended the heat treating. The third insert was made of Q and the fourth one of a modified H-11 composition designated A. The inserts were put in use and their performance monitored. At the time of this writing, the inserts had over 77,000 shots and still working. The K is reported as the least damaged so far, in line with our predictions.

Figure 7.1 shows a schematic of the die with the four identical inserts made of P.G. H-13, Q, k and A steels.

Figure 7.2 is a chart of the die history up to the time of this writing. It indicates all the procedures and operations involved in the use of the inserts, including cleaning, sand blasting and polishing. It also includes operator remarks on the condition of the inserts.

After 36,200 cycles, cavity 14 made of steel K looked the best. After 77,000 cycles cavity 15 made of steel A had to be replaced after it reached a non-repairable damage level. The test is still underway at the time of this writing. Die casting dies that fail in heat checking can last between 20,000 and even 400,000 cycles, depending on the severity of the operating conditions. In-plant testing can therefore take months and even years before conclusion. Note the reported evaluation started in January 1997. It was preceded by six months of design, fabrication and heat treating of the parts. This activity was indeed started very soon after our laboratory testing indicated the superior performance of steel K.

Due to the high cost of the dies and potential disruption in production in case of a failure, most die casters prefer to use only proven suppliers and products. Field testing of critical components is therefore approached with utmost caution. We attribute the willingness of die casters to try the new composition to the well established reputation of NADCA sponsored die materials research work at Case Western Reserve University under the principal investigator, Professor John F. Wallace.

A number of additional in-plant evaluation programs are underway. Some are of proprietary nature and can not be disclosed yet. Others have begun more recently and the results are not available as of yet. Two such programs are described in the enclosed letters. These will continue beyond the current project.

We foresee a gradual transition to the new steel for the next few years. Eventually, even better compositions may become available. It is essential for the competitiveness of the US die casting industry to stay abreast of these developments and constantly implement the best steels and processing methods. It is hoped that the present study will make a significant contribution toward this goal.

Figure 7.1

EJECTOR CENTER INSERT

Figure 7.2

Figure 7.2

		H-13	K	A	Q
DATE	# SHOTS	CAV 13 Results	CAV 14 Results	CAV 15 Results	CAV 16 Results
1/9/97	0	New	New	New	New
1/14/97	5,600	Looks Good	Looks Good	Looks Good	Looks Good
1/21/97	12,100	Minor Solder (opposite of gate)	Signs of solder (opposite of gate)	Signs of solder (opposite of gate)	Looks Good
1/30/97	18,800	Solder building up (opposite of gate)	Solder building up (opposite of gate)	Solder building up (opposite of gate)	Signs of Solder (looks good)
1/30/97	Last Shot - Cores cleaned up in Die Repair-Cavity 16 <i>looked the best</i>				
2/10/97	Start of second run in DCM #101- Cores look good				
2/13/97	6,700	Signs of solder (opposite of gate)	Looks Good	Looks Good	Looks Good
2/17/97 (Last shot in #101)	7,800	Signs of solder (opposite of gate)	Looks Good	Looks Good	Looks Good
2/20/97	Continuation of second run in DCM #115 (900 Ton) - cores were not cleaned up				
3/3/97 (Last shot in #115)	15,900	Signs of solder (opposite the gate)	Looks Good	Looks Good	Signs of Solder (opposite the gate)
3/4/97	Continuation of second run in DCM #101 (700 Ton) - cores were not cleaned up				
3/10/97	21,400	Signs of solder (opposite the gate)	Looks Good	Looks Good	Solder increasing (opposite the gate)
3/14/97	28,300	Signs of solder (opposite the gate)	Looks Good	Looks Good	Solder increasing (opposite the gate)
3/25/97	36,200	Signs of solder (opposite the gate)	Looks Good	Signs of solder (opposite the gate)	Solder increasing more (opposite the gate)
3/25/97	NOTE: The cores were sand blasted and polished when the ejector half was out for a ejector pin fix and put back into production. <i>Cavity 14 looked the best.</i>				
3/27/97	38,600	OK	OK	OK	OK
	(End of second run, 1015 die put in DCM #101)				
Total Number of shots: 18,800 + 38,600 = 57,400 (After 2 runs)					
4/7/97	Start of 3rd Run in DCM # 101 - Cores look OK, minor burning				
4/16/97	9,000	Looks Good	Looks Good	Looks Good	Looks Good
4/23/97	19,600	Solder starting (opposite the gate)	Solder starting Opposite the gate	Solder starting (opposite the gate)	Solder starting (opposite the gate)
4/23/97	Last shot - Putting the 1015 die in DCM # 101				
Total Number of shots after 3 Runs: 57,400 + 19,600 = 77,000 Cavity 15 replaced with a new core. Per Nick Suvak, this core is burnt. (See Core)					
5/9/97	Start of 4th Run in DCM #101 - Cores cleaned up. Look OK

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March 27, 1997

Professor Jack Wallace
 Case Western Reserve University
 400 White Building
 10900 Euclid Avenue
 Cleveland, Ohio 44106-7204

Subject: KDA1 Steel

Dear Professor Wallace:

Over the past two years I have appreciated being involved and actively becoming a Committee member of the NADCA Die Materials Committee. Listening to your presentations and test results of the die steels, General Die Casters has been eager to try the new K H-13 steel.

Recently, Walter Smith suggested we try K on a slide tip. After a few internal discussions, we decided to have a 15" x 15" x 5" replacement slide made with the K. The steel has just arrived at our tool vendor for the start of construction. The die usually runs around 50,000 to 60,000 shots per year and we have an extensive record of the past performance of this tool.

I called Walter to ask him if you might like to know that GDC is making a fairly large slide tip out of K. Naturally, he said you would be interested. As this steel has been a part of your dunk tests, I would like to extend any input or review of this steel in your studies.

If interested, please call me and we can discuss to what extent of information CWRU would like to become involved with.

Currently, I am working with Professor Rajiv Shivpuri on having a full set of inserts CrN. I know OSU will monitor the inserts and castings periodically for wear. What service we can be for CWRU on the K steel needs to be discussed.

Sincerely,

Walter F. Dailey
 Tool Engineer

GDC335WD.SAM

EMPIRE DIE CASTING CO., INC.

Aluminum Alloy Pressure Castings — *Empire* —

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March 24, 1997

Dr. David Schwam
Case Western Reserve University
10900 Euclid Ave.
Cleveland, Ohio 44106-7204

Dear David,

Both John and I would like to thank you for taking the time last Friday to show us around Case Western Reserve University and for sharing your research with us. We were both impressed with the work you have been doing regarding the heat-checking of die steel.

I feel I have an application that would lend itself to this type of research and would be like to invite both you and Dr. Wallace to visit our facility and discuss the possibility of doing an experimental run.

The project would be the testing of your steel in the form of a shot block on a high production part that we run continuously. Our facility has the ability to monitor various machine parameters through the use of a SHOTSCOPE system. I feel that this would be a good test environment because we also employ a RIMROCK automated sprayer and extractor so we can control our machine cycle times much more closely than we could on a manually operated job.

If this sounds at all interesting to you please feel free to call me any time and I will be glad to set up a plant tour.

Once again THANK YOU for allowing us to discuss your research.

Sincerely,

David Palivec

David G. Palivec
Engineer

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