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FOREWORD

This volume contains the High Pressure Technology papers presented at the 2002 ASME Pressure Vessel and Piping Conference held August 4-8, in Vancouver, British Columbia, Canada. These papers were presented in three technical sessions and cover a broad spectrum of topics. The first session, entitled "Methods, Applications and History" was developed by Eric Roll. The second session is entitled "Global Advancement in High Pressure Design and Analysis". This session was developed by Daniel T. Peters. The third and final session entitled "Low Density Polyethylene Applications" was developed by Jan G. M. Keltjens.

The editor would like to acknowledge the technical contributions and expenditure of volunteer time the authors, reviewers, session developers and chairpersons. The editor also wishes to express gratitude to the contributing editors for their significant efforts producing the papers of this volume, as well as for their continuing contributions to the development of the high pressure industry.

Finally, the editor would like to express a special thanks to Ed Perez, my mentor and friend, from whom I have learned a great deal about a multitude of subjects including the high pressure technology. Thank you, Ed, for your patience, instruction, encouragement and example.

Ricky D. Dixon
The DuPont Company

DEDICATION

On behalf of the High Pressure Technology Committee, the editor would like to dedicate this volume to Mr. Leo Picqueur, who passed away at the end of last year. Leo's contributions to the development of the high pressure industry and this series of the High Pressure Technology publications are deeply appreciated.

Ricky D. Dixon
The DuPont Company

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RECENT ADVANCES IN HIGH PRESSURE FOOD PROCESSING EQUIPMENT AND EQUIPMENT REQUIREMENTS TO MEET NEW PROCESS NEEDS

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ABSTRACT

Foods preserved by high pressure processes (HPP) are sold in Japan, the United States, and Europe. HPP technology is used to pasteurize low acid solid and liquid foods such as oysters, hams, and guacamole and to extend refrigerated shelf-life. HPP technology can commercially sterilize liquid and solid acid products such as fruit juices, salsa, and cut tomatoes. Product sales have reached millions of pounds per year. New processes have been developed to sterilize low acid foods using a combination of heat and pressure. Foods at temperatures of 90 to 1000C can be compressed to 600 to 700 MPa for one or more cycles and thus heated uniformly by compression heating in the range of 111 to 121 0C. Decompression brings the product back to its starting temperature for final cooling. This application provides a high-temperature-short-time sterilization process for low acid foods and thus preserves fresh product quality. Commercial HPP foods require rapid cycling of equipment and maximum use of the pressure vessel volume. These requirements have been met in commercial, semi-continuous, liquid food treatment systems. A single 25 liter pressure vessel can cycle 15 times per hour with a three minute product hold at a pressure of 580 MPa. This vessel operating 5000 hours per year can treat over four million pounds of liquid food. Batch equipment designed to cycle over 12 times per hour with a three minute product hold at 680 MPa is under construction. All units manufactured for the HPP treatment of foods use stainless steel contacting parts, potable water as the compression fluid, and are designed to have a safe cycle life of over 100,000 cycles at 580 MPa. Equipment used for the HPP treatment of food must have an up-time in excess of 90% and must be capable of repair and maintenance by food process line technicians. Ease of access and ease of seal and wear part replacement is required. Equipment must meet cleaning and sanitation requirements of the FDA and the USDA if used to treat meat containing products. Pressure chamber volume use in batch systems must be optimized. Even one additional package per cycle at 12 cycles per hour and 5000 hours per year can yield 60,000 additional packages. High cycle rates require automatic package handling systems for loading packages into carriers and for loading and unloading carriers at the pressure vessel.The operation of high pressure food processing equipment must integrate with a specified food packaging and package handling system as it is desirable to have the high pressure processing system as an integral part of the total food processing and packaging system.

INTRODUCTION

Participants in a recent workshop on nonthermal processing methods were asked to develop a list of criteria that would justify the use of high pressure for the preservation of foods as an alternative to preservation by heat, freezing, chemical additives, or dehydration. A review of the products now processed using high pressure equipment yielded a very clear set of criteria. These criteria should be met if a company is to develop a successful line of high pressure preserved food products. The criteria addressed the following issues.

* Product opportunities
* The need for product pasteurization versus sterilization
* The capability of integrating HPP technology into available food processing equipment
* Critical path to commercialization
* Consumer issues
* Manufacturing issues
* Regulatory issues

The product/process combinations selected as case studies were: avocado puree, guacamole, and sliced avocado products pasteurized and distributed refrigerated; oysters shucked and pasteurized using high pressure; and packaged cured hams pressure pasteurized to extend their refrigerated shelf-life. These examples represented high pressure applications in which repeat sales of equipment were realized. Repeat sales indicated that the process filled a commercial need and that the process was economically viable.

Common incentives for the use of high pressure were found among the applications with respect to product needs, critical paths to commercialization, and manufacturing issues. These are identified as true drivers or true incentives to the commercialization of high pressure food processing. It must be understood that these applications use first generation commercial high pressure food processing equipment. These designs are largely based on existing cold isostatic pressing technology. Design improvements to be discussed in this paper may create additional incentives and thus broaden the application of this technology. Also, some disincentives or limitations of the current technology are noted.

RAW MATERIAL AND FINISHED PRODUCT REQUIREMENTS

Raw product materials and the quality of the finished processed product are critical drivers to the purchase of high pressure food processing equipment. The following product factors were identified as needed incentives.

1. Starting raw materials should be relatively low in cost. After treatment the resulting product should yield a relatively high value processed product. Examples are avocados, tomatoes, cut fruits and vegetables, dairy ingredients.

2. Ideal candidate foods and ingredients should be highly perishable materials that cannot be stabilized by competing technologies such as heat without losing their fresh quality. Highly perishable raw products that have a very limited refrigerated shelf-life and thus a limited distribution area as processed products are ideal. High pressure treatment can extend their refrigerated shelf-life and thus allow regional and even national distribution using conventional refrigerated distribution channels. Examples include avocado, tomato, hams, processed luncheon meat, cut fruits and vegetables, seafood.

3. Raw products whereby high pressure treatment gives a unique processing advantage over competing technologies. The best example is the shucking of oysters and other shell fish. Here high pressure allows a clean release of the adductor muscle from the top and bottom shell.

4. Raw products that require pasteurization to inactivate pathogens normally associated with the raw product, but must be marketed with the perception of being fresh. Examples are juices and juice blends, avocado, tomatoes, cut vegetables and fruits, raw seafood, raw or minimally processed animal products.

CRITICAL PATH TO COMMERCIALIZATION

An important incentive to the use of high pressure processing is the ease of transition from existing manufacturing, formulating, and packaging operations to those needed for high pressure treatment. An established market should exist for the product of interest. Existing product preparation procedures, packaging, and distribution methods should be used for the pressure treated product. These are critical factors as the business decision to acquire high pressure equipment can be based on known costs and benefits of an expanded market for the existing product.

Existing or similar packaging structures should be useable in the high pressure process. Many products are defined by or associated with distinctive packages. Even small changes in an existing package to accommodate high pressure processing (as in the case of a package change to allow greater volumetric efficiency in the pressure vessel) may bring sufficient marketing disincentives to cause the abandonment of high pressure processing as a viable technology.

To date most all the companies purchasing high pressure food processing equipment were closely held private firms with a single owner responsible for making the decision to acquire the equipment. The absence of significant regulatory issues; major changes in the product and package; minimal changes due to product flow in the plant; and existing knowledge of manufacturing and distribution costs enabled the owner to acquire high pressure equipment with a reasonable knowledge of the return on investment.

PASTEURIZATION AND STERILIZATION ISSUES

Acid foods (pH below 4.5) can be rendered commercially sterile by high pressure treatment as the pH of the product will block the germination and outgrowth of Clostridium botulinum and other heat resistant spores. Additionally, high pressure can reduce the number of vegetative pathogens to the level needed to guarantee the safety of a refrigerated acid or low acid product for a useful extended refrigerated shelf-life. The useful refrigerated shelf-life is generally controlled by economic factors of distribution or limited by undesirable changes in the chemistry of the product during storage and distribution. In some cases highly pressure resistant vegetative microbes may be injured at time of pressure treatment and may slowly grow back during prolonged periods of refrigerated storage. The process pressure, time, and temperature for optimum shelf-life must be determined in conjunction with the manufacturing practices employed in the preparation of the product.

For commercial success the pressure/time treatments for pasteurization must be short enough (under five minutes) to allow reasonable hourly production rates while significantly reducing the numbers of spoilage microbes (bacteria, molds, yeast) associated with each product produced under good manufacturing practices.

Sterilization of low acid foods is being investigated at this writing using elevated product initial temperatures (up to 100 0C) combined with compression heating using pressures of 600 to 700 MPa. This technology will require a second generation of high pressure processing equipment. At this time pasteurization, in combination with refrigerated distribution, is the optimum application of high pressure to acid and low acid foods.

CONSUMER ISSUES

High pressure treatment of foods should be "transparent" to consumers. That is, the consumer should not need to be aware or even informed of the use of high pressure in the processing of the product. High pressure processing does little to alter the quality of foods. This is an important driver or incentive to the commercial use of the technology. FDA issues are discussed under regulatory issues.

In cases where a product is sold as a "fresh" product, but must be treated to inactivate pathogens, high pressure treatment is considered as a satisfactory pasteurization process. At this time there is a effort to change labeling regulations to allow certain high pressure pasteurized products to be labeled "fresh" much as heat pasteurized, refrigerated milk is recognized as "fresh".

An important incentive for the use of high pressure is the clear absence of any viable competing manufacturing technologies. Thus heat, freezing, ionizing radiation, chemical additives, or other mechanical methods, alone or in combination with refrigeration, must not be economically viable. In the case of chemical additives, consumer issues may dictate the use of high pressure to allow a "clean" label. This is the case with avocado and oysters.

REGULATORY ISSUES

The United States Food and Drug Administration has considered high pressure treated foods, distributed under refrigeration, as equal to non-pressure treated products. Examples are; pressure treated oysters, avocado products, salsa (cut vegetables), juices, and hams. Low acid refrigerated products treated by high pressure to extend refrigerated shelf-life must be proven to be safe under the refrigerated conditions of use. This is the responsibility of the food processor distributing the product.

OPERATING REQUIREMENTS FOR SECOND GENERATION HIGH PRESSURE FOOD PROCESSING EQUIPMENT

Second generation high pressure food processing equipment should provide features such as operating pressures of 600 to 700 MPa; pressure vessel and yoke cycle lives of over 100,000 cycles; and potable water must be used as the compression fluid.

* The equipment must be easily installed into existing food processing lines in existing facilities. Limitations of weight and height must be accommodated. Pressure vessels and yokes designed for slanted or horizontal operation should be available. Horizontal pressure vessels can be conveniently loaded and unloaded to integrate with existing packaging equipment. An extended length pressure vessel for increased capacity is possible. Lower unit area floor loads should be possible with horizontal systems.

* The process system, including pumps, intensifiers, pressure vessels, yokes, closures, and controls must be capable of operating continuously during 20 hour shifts at up to 15 cycles per hour with no component failures. Routine maintenance should be performed by food plant process line personnel and the equipment should be easily and cost effectively serviced and maintained. It should be safe to operate on the food processing floor with people present. Equipment, including control electronics, should be easily sanitized using conventional food processing line sanitizers.

* There is increasing interest in the use of elevated temperatures in conjunction with pressure to sterilize low acid foods. At this time combinations ~~of~~ temperatures in the range of 120 0 C with pressures in the range of 680 MPa have been shown to sterilize low acid foods in a few minutes time. The "Holy Grail" of high pressure food preservation is to sterilize foods with a minimum of heat. Compression heating allows the temperature of a food in the range of 100 0 C to be elevated to 120 0 C uniformly and rapidly without concern for heat transfer. Decompression returns the product to its 100 0 C starting temperature for further conduction cooling.

* Process controls must meet hazard analysis of critical control points (HACCP) requirements. System controls should provide ease of set-up of processing temperature, pressure, dwell time at pressure, and capability of pulsing if needed. Pressure, temperature, and time should be recorded in hard copy.

DESIGN NEEDS TO MEET IDENTIFIED OPERATING REQUIREMENTS - FAILURE ANALYSIS

A. Introduction

This section highlights the weakest links existing in current high pressure processing systems of interest to the food processing industry. Reliability as used in this analysis is not safety related in that component failure, while under pressure, can be designed to minimize safety hazards. Indeed, all systems and all sub-systems and components subjected to pressure are expected to be designed to fail in a safe manner. However while a high pressure line can rupture and be shielded to prevent injury or damage, the concern here is that the manufacturing process would be brought to a halt while the line was being replaced. The loss of system productivity may be many times the cost of replacement of the failed line. Thus, reliability as used here is defined as absence of lost manufacturing time due to component failure.

Current designs may function for several hundred cycles at 680 MPa, however equipment as a system must be able to operate for 20 hours at 15 cycles per hour (total 300 cycles) without failure. While it is acceptable to maintain the system every 20 hours, repairs should be minimized. Replacement of wear parts may be acceptable every 100 to 120 hours provided the cost of new parts and their installation costs are modest (gaskets, seals, disposables).

High pressure food processing systems may be separated into sub-systems and components for the purpose of operating reliability analysis. These can be grouped by expected or projected reliability based on failure experience while operating at 680 MPa. Components with low reliability are those needing design efforts to qualify them for second generation food processing systems.

There is always a balance between the useful life of a component and its cost including inventory cost and cost of installation. Here it must be assumed that any part or component, to be acceptable for food processing applications, must have a minimum operating life of twenty hours regardless of the number of cycles taking place in this period (see note *). Low cost disposable parts may be acceptable if installation can be included in the routine maintenance cycle every 20 hours.

* An unique example is pressure pulsing. Pressure pulsing with two or more short pressure cycles may result in more effective microbial inactivation than a single longer pressure cycle. Pulsing

technology could result in the use of over 900 complete compression-decompression cycles in 20 hours.

B. Components With Expected High Reliability (Long Times Between Failure)

General Equipment: Support structures, control housings, utility lines, low pressure pumps, hydraulic operating components, tracks, safety shields, jackets, finishes, low pressure valves, electronic data processing systems, connecting wires, mechanical pressure gauges, electronic control systems, switches, other circuit components.

Pressure Vessel (leak before break): Liner (except for seal area), outer pressure vessel, pressure vessel moving and positioning system.

Yoke: All parts of yoke, yoke moving and positioning system.

Low Pressure Pump: Pump, oil reservoir, controls, valves, input transducers, liquid lines, oil, filters, micro-switches.

C. Components With Expected Reasonable Reliability (Failure can be Anticipated and Cost Effective Repairs or Replacements Made Without Sacrificing Operating Up-Time)

Vessel Closure Plates: End plates (but not sealing system), through packing glands carrying wires for internal temperature, pressure, and other internal data measuring transducers. Automated end plate opening, positioning, and closing components.

High Pressure Lines and Fittings: All lines and fittings connecting intensifier, pressure vessel, pressure transducers, rupture discs.

Carriers: Package carriers, automated carrier handling systems.

Water: Water purification systems, filters, temperature control systems.

D. Components Requiring Improvement Due to a High Probability of Failure Within a 20 Hour Operating Period

High Pressure Valves: Pressure control and relief valves, check valves.

Seals: Pressure vessel sealing systems, pressure vessel sealing surfaces, intensifier seals, packings.

Capillary Tubing: All high pressure capillary tubing, connectors.

Intensifier: Intensifier system is inherently subject to wear. Better designs are needed.

CONTROL SYSTEMS

Food processing systems must have process controls which provide a permanent record of process conditions as a function of time for each batch of food treated. Expert panels have recommended that pressure and temperature be recorded with an accuracy of + 1/2%. Traceable pressure and temperature equipment must be used to validate and monitor the process.

The use of process temperatures up to 121 0 C (250 0 F) have been proposed to increase the rate of microbial inactivation during pressure treatment. Food and Drug regulations require that every element of food be exposed to the proposed process temperature during the pressure treatment. Since the compression of packaged foods in water can increase the temperature of the water and food by 14 0C (25 0F) or more for a pressure increase of 100,000 psi (680 Mpa), care must be taken to reproduce chamber loading and pressure increase for each cycle. It is generally not possible, nor is it desirable, to measure the internal temperature of packaged food during treatment.

Pressure, temperature, and time outputs must be capable of being recorded to achieve a permanent record. These records are held until the food is consumed so that any microbial problems can be traced to the process conditions.

CONCLUSION

High pressure treatment of foods in semi-continuous pumped systems or in batch isostatic presses represents a potentially large market for high pressure equipment. This market will not develop unless the high pressure food treatment system is capable of very high cycle life at 100,000 psi (680 MPa), is reliable during daily operation, and is easy to inspect maintain, and repair by the user.

The system must be safe to operate and must be fully automated so that it can be operated by food processing employees. Controls must provide and accuracy of + 1/2% and outputs must be available in a form that can be stored for later reference and review by Food and Drug Officials.

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Intensive Quenching Theory And Application For Imparting High Residual Surface Compressive Stresses In Pressure Vessel Components

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ABSTRACT

An alternative method for the hardening of steel parts has been developed as a means of providing steel products with superior mechanical properties through development of high residual compressive stresses on the part surface, and involves the application of intensive quenching during heat treatment. This processing method, commercially patented under the name IntensiQuench^{SM}, imparts high residual compressive stresses on the steel surface, thus allowing for the use of lower alloy steels, reduction or elimination of the need for carburization and shot peening, and providing for more cost-effective heat treating. Intensive quenching also provides additional environmental benefits, as the process uses plain water as the quenching media in contrast to traditional heat treatment practices which typically employ hazardous and environmentally unfriendly quenching oil. This paper presents an overview of the theory and application of intensive quenching, as well as provides experimental and computational data obtained for a variety of steel products. Also presented will be results of computer simulations of temperature, structural and stress/strain conditions for a typical pressure vessel during intensive quenching.

THE BASICS OF INTENSIVE QUENCHING

There are several different quenching techniques used in common practice today, including direct quenching, time quenching, selective quenching, etc. The selection is based on the effectiveness of the quenching process in considering the materials, parts, and quenching objectives (usually high hardness with acceptable distortion). In all cases, the quenching process is controlled to prevent a high cooling rate when the material is in the martensite phase. This rule is based on the belief that a low cooling rate in martensite will avoid high tensile, residual stress, distortion, and the possibility of part cracking.

Extensive research conducted in the Ukraine by Dr. Nikolai I. Kobasko has shown that avoiding a high cooling rate when material is in the martensite phase is not always necessary or optimal to obtain the best properties. His studies showed that a very high cooling rate within the martensite range would actually prevent quench cracking, if done correctly. This phenomenon was discovered first by laboratory experiments and then was supported by computer simulation (References 1 and 2). A large number of field experiments on a variety of steel parts validated both the theory and the computer simulation (References 3 and 4).

Figure 1 shows experimental data obtained for a cylindrical specimen made of a low alloy steel with a diameter of 6 mm (about 0.25"). The bell-shaped curve clearly illustrates the general effect of the cooling rate within the martensitic phase on crack formation: the probability of quench cracking is low for both slow and *very rapid and uniform* cooling. This high cooling rate regime is termed "intensive quenching". The curve also shows that once quenching is in the "intensive zone" or above, the benefits of using this process – high hardness *and* low distortion -- will be attained. One cannot quench "too fast" because once the surface temperature of the part reaches the quenchant temperature, the part simply cannot cool any more quickly; cooling is limited by the ability of the part to conduct the heat energy from the core to the surface.

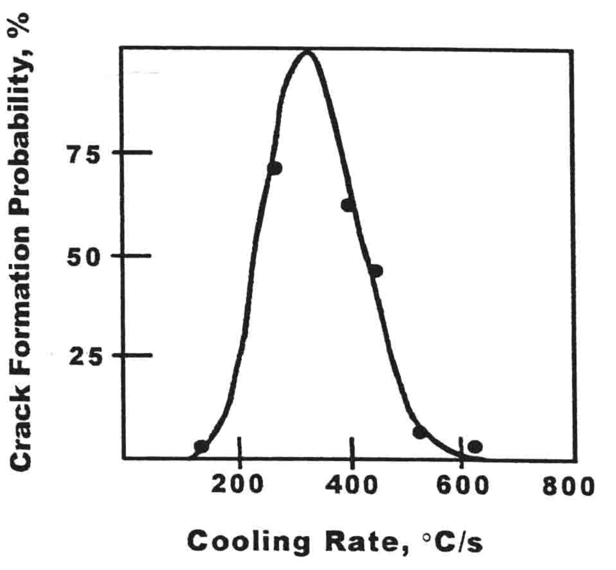


Fig. 1 Effect of Cooling Rate on Probability of Cracking

__Mechanism__

Imagine a steel part with a varying thickness (Figure 2). During conventional quenching, the martensite forms first in the thinner section of the part since this section cools faster and reaches the martensite range earlier than the thicker section (Figure 2a). The martensite specific volume is greater than the specific volume of the remaining austenite. Therefore, the thin section expands while the thick section of the part continues contracting due to cooling until it too transforms to martensite. This creates stresses resulting in the distortion and possible part cracking.

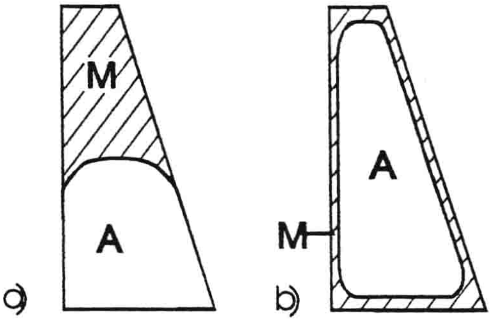


Fig. 2 Martensite Formation During Quenching
a) Conventional b) Intensive

Now imagine that the same steel part is cooled very rapidly and uniformly. In this instance, the martensite forms simultaneously over the entire part surface, creating a hardened "shell" (Figure 2b). Dr. Kobasko's research showed that this uniform, hardened shell creates high compressive stresses resulting in lower distortion and lower probability of cracking.

Compressive Stress Formation

To simplify a mechanism of the stress formation in the part, assume that the part consists of only two sections: a "surface layer" and a "core." (It would be more accurate to consider the part as a series of concentric layers, like layers of an onion, where the heat and the phase transformation are "transferring" from layer to layer.) Now assume that the part's "surface layer" consists of a set of "segments" joined together by "springs" to form an elastic "ring" (Figure 3). When the whole steel part is austenitized (heated and held above Ac_3 temperature) before quenching there is no tension in the "springs" and there are no stresses between the "segments" (σ=0, see Figure 3a). During quenching, the surface layer cools rapidly resulting in the contraction of the "elements." To compensate for the contraction of the segments in the surface layer during cooling, the "springs" expand simulating the development of tangential (hoop) *tensile* thermal stresses (see Figure 3b).

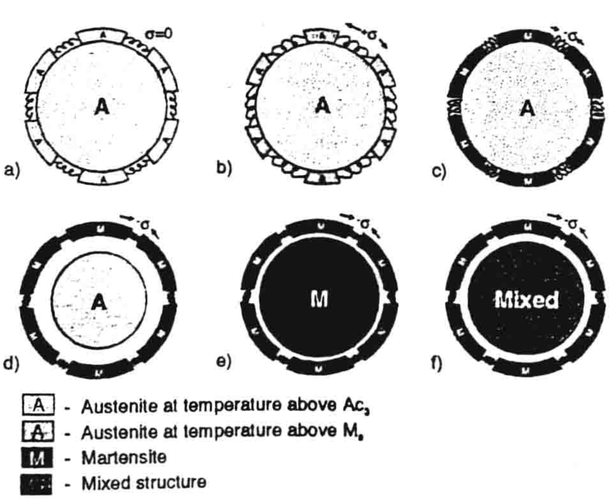


Fig. 3 Surface Stress Conditions During Intensive Quenching

When the surface layer reaches the martensite formation start temperature, Ms, the austenite in the surface "segments" transforms into martensite (see Figure 3c). The martensite specific volume is greater than that of austenite. This results in the expansion (swelling) of the surface layer "segments", causing the "springs" to contract. The contraction of the springs illustrates the development of surface *compressive* hoop stresses.

It is important to note that during intensive quenching, the part surface layer reaches the martensite start temperature M_s so quickly that the part core is still very hot (practically at the initial austenitizing temperature). (This is in contrast to conventional quenching, for example marquenching, when the part core temperature may be just above the M_s temperature at this period of time.)

While the martensitic structure is forming in the part surface layer, the part's austenitic core continues to cool down to the M_s temperature, shrinking in size as it cools (Figure 3d). We call this core thermal contraction "pre-phase transformation shrinkage." As the core shrinks, the strong martensitic shell maintains the part's initial size with low distortion – almost as though a "die" has been built on the outer shell of the part. The shrinking (cooling) austenitic core draws the martensitic surface shell toward the part center increasing the surface hoop *compressive* stresses (with the "springs" between the surface layer "segments" contracting). Note that in a real quench the material does not "break" between the shrinking austenitic core and the fixed martensitic "shell" (as shown on Figure 3d). This is because the hot austenite is in a "plastic" state; and when stresses between the "surface" and "core" sections of the part exceed the austenite yield strength, the austenite deforms to maintain part integrity within the shell.

If intensive quenching continues further, then within a short time (in a matter of seconds), the martensite starts forming in the part "core," resulting in the core swelling (see Figure 3e). The expanded part core pushes the part surface layer back from the part center resulting in diminution, but not elimination of the high surface compressive stresses. (Put another way, the distance between the surface layer "segments" increase, resulting in the expansion of the "springs" and the lowering of the compression in the surface shell). The surface residual stresses are still compressive even in a through-hardened part because the size of the expanded, martensitic core is actually smaller than the size of the initial, hot austenitic core. In other words, the steel's *pre-phase transformation shrinkage* (of the cooling austenitic core) offsets the following phase transformation *expansion* in the final, martensitic core.

At some point in time, the surface compressive stresses reach their maximum value. It happens just before martensite starts forming in the core. *The key element of the IntensiQuench[SM] technique is to "interrupt" the rapid, uniform cooling of the part's "shell" when compressive stresses in the part's surface are at their maximum.* The "interruption" is done by simply removing the part from the intensive quench. As the cooling rate of the part "shell" slows, the part "core" will also begin cooling more slowly and the martensite phase transformation advance may slow or cease entirely if the part is thick enough (over approximately one inch). If the martensite formation ceases, the remaining austenite in the core transforms into intermediate phases, such as bainite, ferrite and pearlite (See Figure 3f). Since this mixed "core" structure has less specific volume than a "pure" martensite core (as discussed above), the interrupted quench results in a higher level of surface residual compressive stresses when compared to the through-hardened version (see Figure 3e). The precise time for interruption is predicted by the IQ Technologies computer software model. Usually, there is a window of several seconds to move from each stage of the intensive quench process; the thicker the part, the "bigger the window." As such, IntensiQuench[SM] is robust and practical for production environments.

It is important to note that the ability of intensive quenching to create residual compressive surface stresses, even when the part is through-hardened, is in stark contrast to conventional quenching, where residual surface stresses are usually tensile or neutral. This is because in conventional quenching the part cools several times slower than in intensive quenching, and the temperature gradient throughout the part is small. Therefore, in standard quenching, the part core temperature is just above the M_s temperature when martensite starts forming in the part surface layer. In contrast, in intensive quenching the part core is very hot at the same moment of time (Figure 3c). The pre-phase transformation shrinkage of the core in this case is negligible compared to intensive quenching (see Figure 3d) and it does not offset the subsequent core expansion. This is the metallurgical "key" to the process. In non-intensive quenching, part core expansion is actually greater than the pre-phase transformation thermal shrinkage. Therefore, after conventional quenching, the swelled core pushes apart the surface "segments" creating tensile stresses on the part surface (the "springs" between the "segments" expand, as shown schematically in Figure 4. This is why many conventionally quenched parts are very "unstable" and may crack if not tempered soon after quenching.

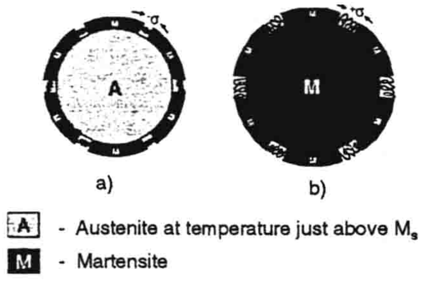


Fig 4. Surface Stress Condition During Conventional Quench

Curve I on Figure 5 provides an additional illustration of the dynamics of the surface stress conditions during intensive quenching over time with a "mixed core," while Curve II on the same figure illustrates the dynamics of the "surface" stress conditions during intensive quenching over time with a "martensitic core" structure. In both cases the initially created surface compressive stresses are of such magnitude so as to be able to remain in residual compression even after subsequent transformation and expansion of the core. In contrast, the lower level of surface compression created during the initial quenching stage using a standard immersion practice is insufficient to withstand the subsequent core transformation, often resulting in a net neutral or tensile residual surface stress (Curve III on Figure 5).

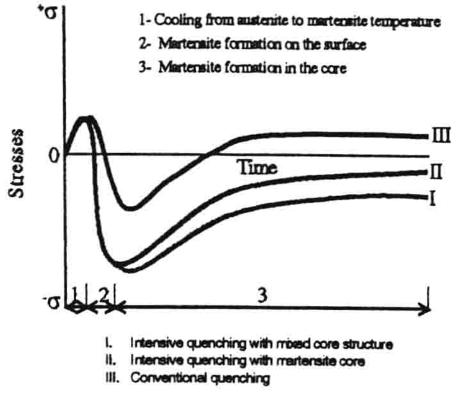


Fig. 5 Surface Stress vs. Time

In a "real" part, one with more than "a surface layer" and "a core," this phenomenon is repeated in layer after layer of the part until the entire part is cooled below martensite formation finish temperature, M_f. In actual parts, the austenite to martensite transformation takes place in a sequence of concentric layers much like layers in an onion (Figure 6). Once the "shell" is cool and is in deep compression, the intensive quench is "interrupted."

Once the intensive quench is interrupted, the "layers" beneath continue to cool by conduction. Since heat conduction within a solid is very uniform and relatively fast, the uniform cooling of the part continues (even after the "interruption" of the intensive quench), resulting in a mixed structure in the part core with residual compressive stresses on the surface. Each concentric layer of the part goes through the same thermal *shrinkage* (from cooling austenite) and the same phase transformation *expansion* (from forming martensite or other hardened phases) until the part is fully transformed.

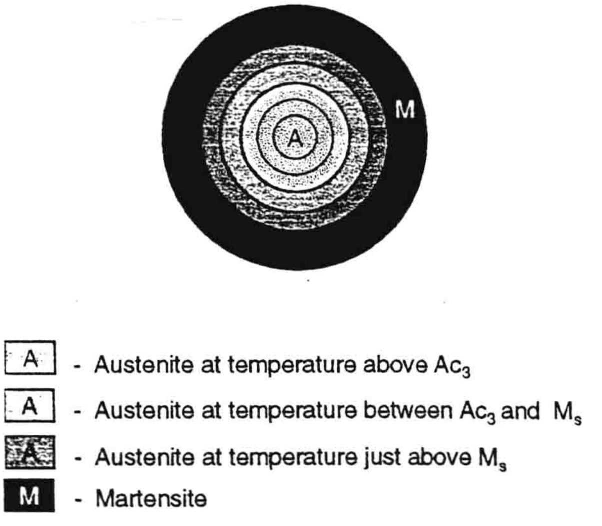


Fig. 6 Part Structure Concentric Layers

The intensive quenching phenomenon of high surface compressive stress in through-hardened parts and in the parts with the mixed structure in the core is confirmed by the results of detailed computer simulations of part thermal and stress/strain conditions, and more importantly by experimental data and case studies (detailed in the later sections of this paper). Numerous laboratory and field experiments have shown that the strength of the final part is related to the speed of the quench (or the rate of external heat transfer). The increased strength and higher surface compressive stresses due to intensive quenching help to eliminate quench distortion and enhance the durability (service life) of machine parts and tools (References 5 and 6).

Optimum Hardened Depth.

Analysis conducted by Dr. Kobasko shows that the "optimum hardened depth" of the "shell" corresponds to the "maximum" compressive surface stress, and is a function of the part dimensions and part geometry. For best results from intensive quenching, the steel alloy (and its related "hardenability") should be selected in consideration of the part's geometry to ensure that hardening occurs to the optimum depth. The higher alloy or deeper hardenability steels are not always the best choice for the intensive quench to create high compressive surface stresses and still be able to interrupt the quench at that point to slow the transformation of the core. Computer modeling provides a high level of accuracy in predicting and determining this optimum depth.

The intensive quenching method will provide an optimum combination of high residual compressive surface stress; high strength and wear-resistance due to high surface hardness; a quenched layer of optimum depth; and a relatively soft but properly strengthened core (Reference 4). This combination is ideal for applications requiring high strength and resistance to static, dynamic, or cyclic loads. Dr. Kobasko also demonstrated experimentally that by applying the intensive quenching method, the desired properties of the part could be obtained using less expensive steels (steels containing two or three times less alloying elements than conventional alloy grades). The process has been used successfully on solid parts with section sizes up to 40 inches.

Since water is the best intensive quenchant due to its high heat-extracting index, another benefit of the intensive quenching process is the elimination of oil, salt and other potentially hazardous quenchants.

IQ PROCESS COMPUTER MODEL

Development of an intensive quenching process begins by analyzing the thermal and stress profiles within the part during quenching using a finite element approach. Dr. Kobasko and his colleagues developed a two-dimensional computer model to conduct these analyses (Reference 5). This model includes a non-linear, transient heat conduction equation and a set of equations for the theory of thermoplastic flow with kinematic strengthening under the appropriate boundary conditions on the part's surface. Numerous laboratory and field experiments have been used in the validation of this computer model.

A similar but three-dimensional software package, DANTE[1], was developed in the US on the collaborative research program managed by the National Center for Manufacturing Science (Reference 10). However, in contrast to the computer model described in (Reference 5), the DANTE software does not calculate heat transfer boundary conditions on the part surface; rather, DANTE involves the application of heat transfer coefficients or fluxes at part surfaces. An accurate characterization of these conditions is a key element to the accuracy of such calculations. The analysis results that are presented in this paper are based on the DANTE software package , with boundary conditions determined using Dr. Kobasko's computer model.

For an evaluation of the potential for application of the intensive quenching method to the heat treatment of pressure vessels, three possible intensive quenching methods were simulated and compared with predicted simulation using standard water quenching techniques. The pressure vessel under evaluation was chosen as a generic open end cylinder (blind end vessel) with OD = 457.2 mm (18.0 in.), ID = 228.6 mm (9.0 in.), and length = 1270 mm (50.0 in.) made of 4340 steel. The normal manufacturing route for this type of pressure vessel involves forging of the vessel shell, preliminary machining, heat treatment, and possible final machining. The cylinder is typically heat treated by horizontal immersion, with agitation applied to the internal cavity to avoid excessive build-up of vapor. Application of the intensive quenching process is designed specifically to impart compressive stresses onto the affected surface regions of the heat treated pressure vessel.

To model both the standard and intensive quenching processes, a 2-dimensional mesh was prepared for an axisymmetric section (one half) of the 1270 mm (50 in.) long "blind end" pressure vessel previously described. The 2D mesh is shown in Figure 7, and contained 3056 nodes with 2850 quadrilateral elements. Both models assumed a uniform starting temperature of 900 C and a completely austenitic microstructure that was stress free. Quenching was simulated through the application of surface heat transfer coefficients that were supplied by IQ Technologies Inc. for both the standard immersion and intensive quenching processes [17, 18]. The DANTE[1] material model was used for the quench hardening simulations, using the ABAQUS[2] finite element solver. In this application, intensive quenching used highly agitated water as the quench medium. An extremely high level of water flow is necessary to eliminate not just film boiling but also nucleate boiling during quenching so that intimate contact between the pressure vessel and water is maintained. Based on experiments conducted by IQ Technologies Inc., surface heat transfer coefficients of 20 to 40 $kW/(m^2{*}C)$ are achieved during intensive quenching. For comparison, peak heat transfer during water quenching is on the order of 5 $kW/(m^2{*}C)$ and the average is less than 1 $kW/(m^2{*}C)$.

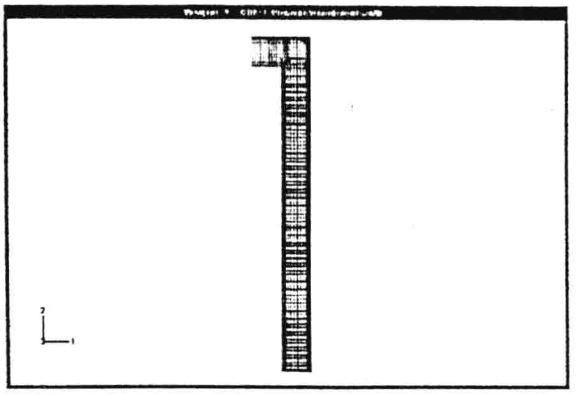
Two simulated heat treatments for the pressure vessel were evaluated; one using the current "standard" immersion water quench, and the other three using a customized intensive quenching process designed to provide a relatively equal heat flux density between inside and outside surfaces. Processing data for the simulation of each heat treatment is presented in Table 1.

Table 1.
Process Data Used for Simulations.

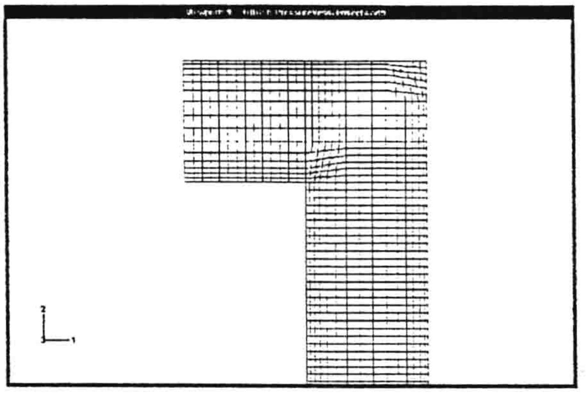
	Standard	IQ Case 1
Aust. Temp	900 °C	900 °C
Quenchant Ambient	30 °C	30 °C
Orientation	Horizontal	Horizontal
Exterior Heat Transfer	700 W/m^2K	7000 W/m^2K
Interior Heat Transfer	700 W/m^2K	20,000 W/m^2K
Quench Time	40 Minutes	32 Minutes

Resulting microstructure and stress states were then examined in each simulated case.

[1] DANTE is a registered trademark of Deformation Control Technology, Inc., Cleveland, OH.
[2] ABAQUS is a registered trademark of HKS, Inc., Pawtucket, RI.



a) Overview of Axisymmetric Mesh



b) Close-up of Closed End Illustrating Surface Mesh Refinement

Fig. 7 Finite Element Model Mesh Developed for Pressure Vessel Heat Treatment Evaluation

Standard Quench – Microstructure and Stresses

Simulation of the standard horizontal immersion quenching of the pressure vessel produced the time-temperature history shown in Figure 8. Note the maximum thermal gradient from surface to center is about 330 °C at about 2 minutes into the quench.

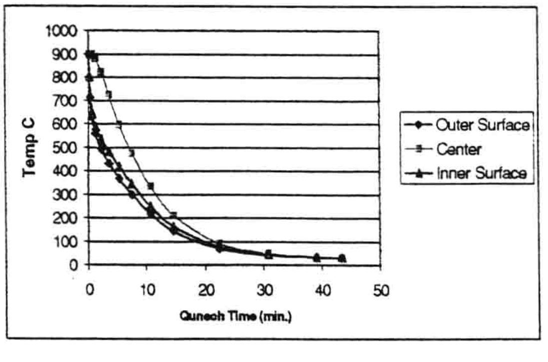


Fig. 8 Time-Temperature History for Oil Quenched Pressure Vessel Section

Figure 9 shows a contour map of the resulting hoop stress through the vessel cross-section upon completion of the immersion quench (stress in MPa). Note that the difference between the interior and exterior surface areas contributes to a slight difference between the inner and outer surface temperature profiles, which is in turn manifested in a difference between resulting surface hoop stresses – with the interior displaying lower residual surface compression than the outside.

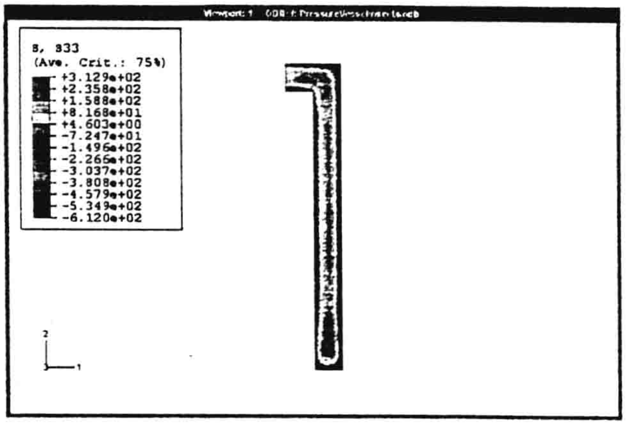


Fig. 9 Profile of Residual Hoop Stress in Pressure Vessel Cross-Section after Oil Quench (units=MPa)

Figure 10 shows a time history of both residual hoop stress and transformation behavior for both the vessel surfaces and center. Note that both the inner and outer surfaces are predicted to be under residual compressive stresses, and that the time at which these stresses reach their maximum is well after completion of transformation.

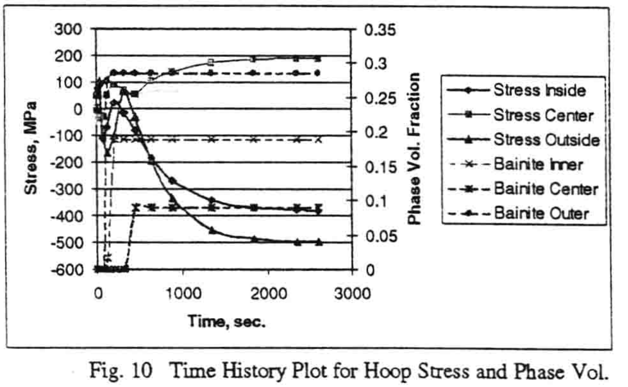


Fig. 10 Time History Plot for Hoop Stress and Phase Vol. Fraction for Surface and Core Areas within the Pressure Vessel during Oil Quenching

As the history plot in Figure 10 shows, the majority of the compressive surface stresses are generated after transformation of the core, and are developed primarily through thermal shrinkage of the core. This is standard behavior in large or thick-section parts. The final microstructure is predicted to be mostly ferrite-pearlite and bainite with no martensite.

Intensive Quench – Microstructure and Stresses

In the Case #1 intensive quenching scenario, the process designed and proposed for this application involved application of a highly agitated salt water solution to provide continuous convective cooling of the outside surface, calculated as 7 kW/m^2 K. On the interior of the pressure vessel, this same solution provides a higher average heat transfer (on the order of 20 kW/m^2 K) due to nucleate boiling. There are three principal regimes of boiling with respect to surface heat transfer behavior: film boiling, nucleate boiling, and free convection [19]. During nucleate boiling, which typically occurs within about a 30°C temperature range, bubbles form at nucleation sites and continually separate from the quenched surface. This separation induces considerable fluid mixing and subastantially increases both the surface heat flux and consequent heat transfer coefficient. Directed quenchant agitation, as well as certain chemical additives, are employed in the intensive quenching process to maximize this effect. Specifically, this IQ process was designed to provide a relatively equal heat flux density between inside and outside surfaces. As shown in Figure 11, the resulting surface to center thermal gradient is now substantially higher; on the order of 700 °C for the interior to center, and 800 °C for the outside surface to the center.

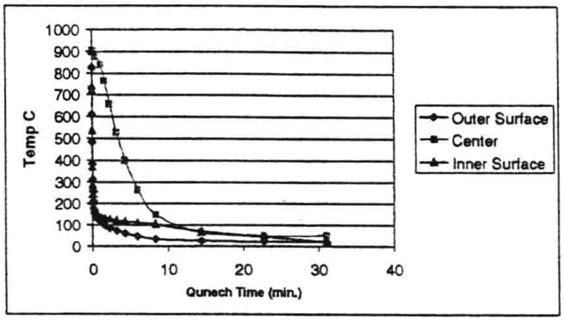


Fig. 11 Time-Temperature History for Intensively Quenched Pressure Vessel Section, Illustrating Extreme Thermal Gradient

The time history for residual hoop stress and transformation behavior for this quenching scenario is shown in Figure 12. In this case note that the surface phase transformation is martensitic, and that this transformation is completed well before the bainite transformation in the center even begins. The core remains in a fairly neutral stress state until it transforms to bainite (30%) and ferrite-pearlite (70%), at which time it briefly expands generating tensile stresses in the center and very slightly reducing the compressive stresses at the surface. However, the bulk of the surface compressive stresses remain intact due to the inherent strength of the martensite phase, even during subsequent cooling with the associated thermal shrinkage.

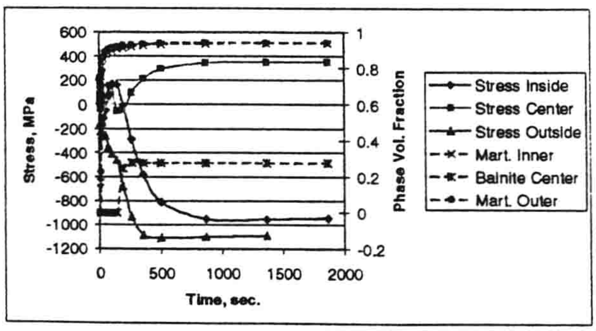


Fig. 12 Time History Plot for Stress and Phase Vol. Fraction for Surface and Core Areas within the Pressure Vessel during Intensive Quenching

Figure 13 shows the final predicted metallurgical phase fractions present in the cross section at the end of quench, with the corresponding final hoop stress profile shown in Figure 14. Again, the primary feature is the predicted predominant martensitic surface layer and much higher level of beneficial surface compressive which extend deeper into the cross section.

Comparison of Quench Simulation Results and Implications

A more detailed comparison of the standard and intensive quenching simulation results as applied to heavy section steel components reveals several important differences, benefits, and limitations.

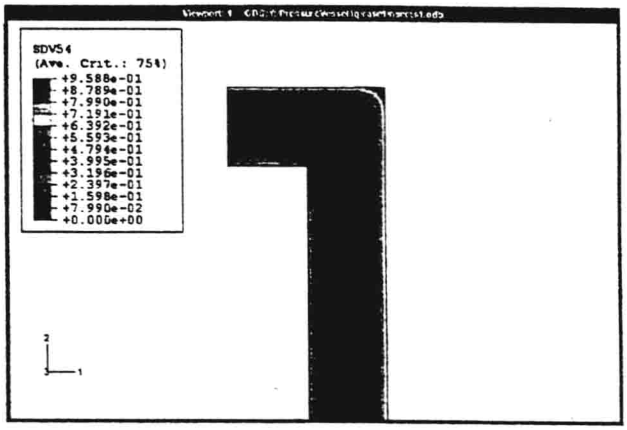


Fig. 13 Contour Plot of Martensite Phase Vol. Fraction in the Pressure Vessel Section at the End of Intensive Quenching

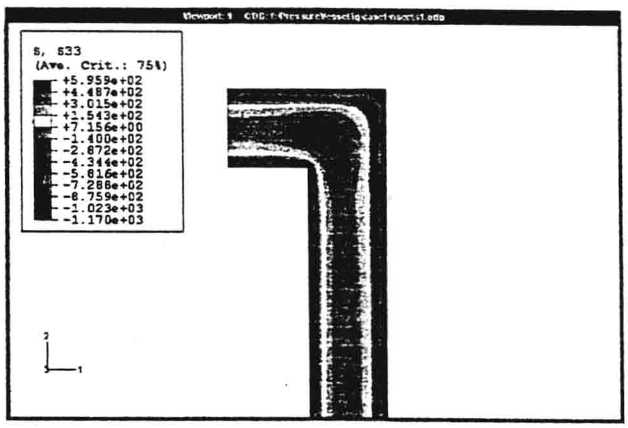


Fig. 14 Profile of Residual Hoop Stress in Pressure Vessel Cross-Section after Intensive Quench (units=MPa)

First, in heavier section components the thermal gradients generated in both quenching techniques are more localized at the component surface, though in intensive quenching the magnitude of 700° to 800° C is substantially higher than the magnitude of about 100° C for oil quenching. The ability to complete the surface martensitic transformation prior to the initiation of the core bainite-ferrite-pealite formations in intensive quenching provides important resistance to the later damping of the resulting surface compressive stresses due to subsequent transformations in the core.

The general propensity for the generation and retention of surface compressive stresses is greater in larger, thicker steel sections than in thinner ones. This is because in general, the thermal gradients created in the larger sections are insufficient to create martensite in the core to a degree which would counteract the surface compression created by the formation of martensite at the surface. Thus the use of the intensive quenching technique generally has a greater benefit in thinner than in thick sections, though it is still beneficial in thick sections as well. In these thicker sections, which often retain some degree of surface compression after standard quenching, intensive quenching will impart even greater surface compressive stresses, as indicated in the comparative plot shown in Figure 15.

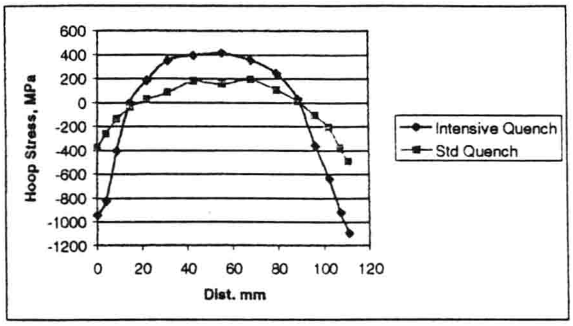


Fig. 15 Comparative Stress Profiles through the Vessel Section for Both Quench Scenarios

The graph in Figure 15 indicates an almost 2x's increase in surface compressive stress for the intensively quenched section as compared to the standard quench. However, due to the limiting effect of thermal conductivity, the presence of compressive stress in general (for both the intensive and standard quenching) is limited to a depth of about 20 mm.

An overall improvement in surface and through-hardness results is also predicted by employing intensive quenching on the large section pressure vessel. The plot presented in Figure 16 illustrates the predicted hardness profiles for both the standard and intensively quenched pressure vessel sections. The benefit of intensive quenching is the achievement of martensite on the surface and a higher bainite content in the core as opposed to bainite-ferrite-pearlite throughout the oil quenched cross section.

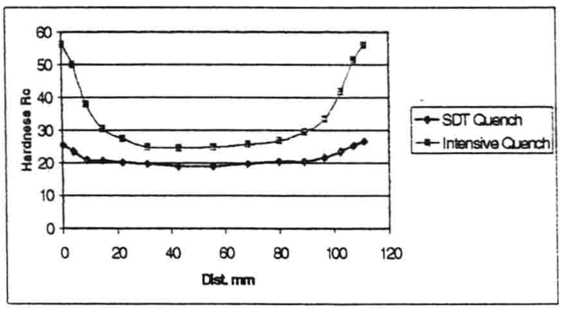


Fig. 16 Comparative Hardness Profiles (Predicted) for Both Quench Scenarios

The general implications and potential benefits of employing the intensive quenching technique to pressure vessel sections involve potential enhancements in service performance. It is well known that compressive hoop stresses are beneficial in applications where fatigue life of a component is important, as they are in the case of a pressure vessel. The enhanced residual surface compressive stresses produced by the intensive quenching process would provide improved resistance to fatigue during pressurization and depressurization cycles and enhance vessel life. In addition, though not evaluated directly in this study, the application of intensive quenching has also been shown in several component trials to markedly reduce part distortion, as the rapidly developed and very high strength martensitic shell acts to quickly "lock-in" the part shape. Thus in the areas of both improved fatigue life and minimal part distortion in heat treatment (leading to a reduced need for final machining), the intensive quenching technique displays promising potential for application in the production of pressure vessels.

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