



Case Study

U.S. Chemical Safety and Hazard Investigation Board

NDK Crystal, Inc., Belvidere, IL High-Pressure Vessel Rupture

December 7, 2009

One Public Fatality, One Public Injury

No. 2010-04-I-IL

KEY ISSUES

- Pressure Vessel Design and Material Selection Requirements
- Learning from Previous Incidents
- Inspections

INSIDE

- Introduction
- Process Discussion
- Incident Description
- Consequences
- Metallurgical Analysis
- Findings
- Recommendations



This case study examines the violent rupture of a 50-foot pressure vessel used to produce synthetic crystals at the NDK Crystal facility in Belvidere, Illinois. The vessel, located in an enclosed building, generated several projectiles during the explosion that resulted in one fatality and one injury to members of the public, and significant property damage to NDK Crystal and to an adjacent business.

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1.0 INTRODUCTION

1.1 NIHON DEMPA KOGYO (NDK) CO., LTD.

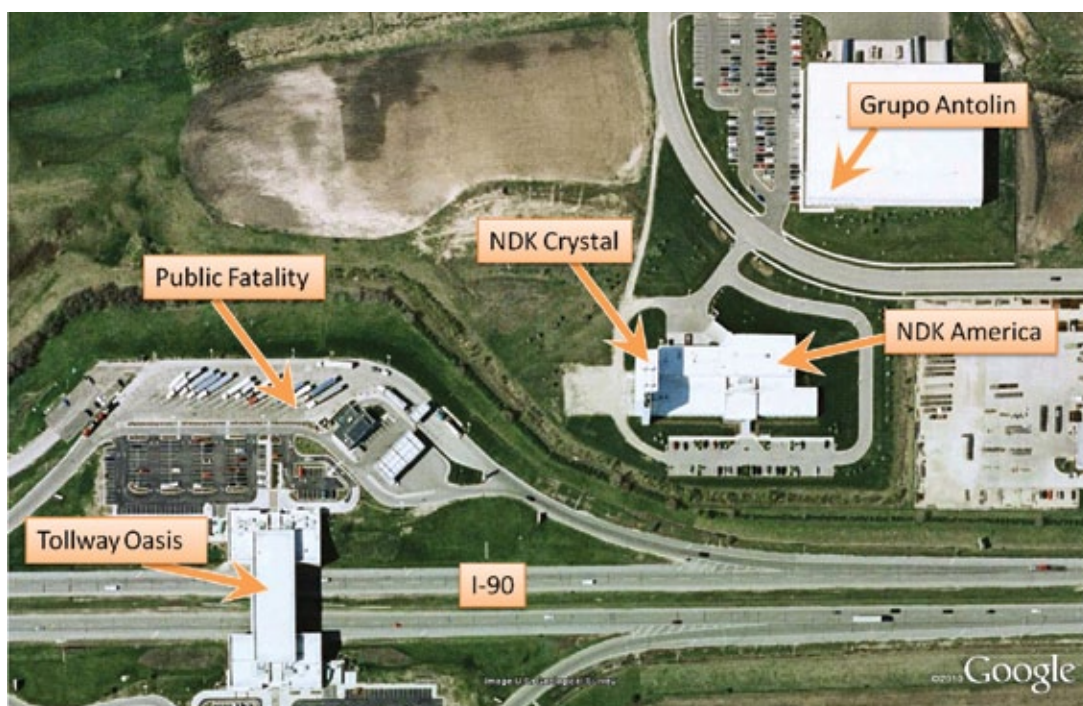
Founded in 1948 in Tokyo, Japan, Nihon Dempa Kogyo CO., LTD. (NDK) began producing synthetic crystal products and over time, has expanded its production and sales facilities to the United States, Europe and Asia. NDK produces and sells crystal-related products such as oscillators, ultrasonic transducers, and synthetic quartz. These products are commonly used in cell phones and wireless internet devices.

1.2 FACILITY DESCRIPTION

The NDK Crystal facility in Belvidere, Illinois is a synthetic quartz manufacturing facility that was built in 2002 and began operation in 2003. The facility is located in a light industrial area adjacent to Interstate 90. The NDK Belvidere facility is five stories tall and houses eight vertical pressure vessels called autoclaves¹ (cover photo). The vessels are 50 feet tall and made of over eight-inch thick steel. NDK Crystal shares the property with NDK America, Inc., the sales and marketing portion of the company (Figure 1). The Belvidere facility is the only NDK production facility in the United States.

FIGURE 1

Overhead view of NDK facilities, Grupo Antolin (neighboring company), and I-90 Tollway and Oasis rest area.



¹ An autoclave is a heavy, heated vessel assembly commonly used for sterilization or conducting chemical reactions under high pressure and temperature.

1.2.1 Vessel Description

The eight NDK vessels were designed and built for crystal growing operations that require a vessel material and thickness to withstand high operating pressures. The vessels consist of a 48-foot long cylindrical shell and a 2-foot thick closure head that operators clamp to the top. The cylindrical wall is 8.1 inches thick and the top and bottom of the vessels are significantly thicker than the vessel walls at 18- $\frac{1}{4}$ inches near the lid and 16- $\frac{1}{4}$ inches at the base (Figure 3). Engineered Pressure Systems, Inc. (EPSI) designed and engineered the vessels and Sheffield Forgemasters (Sheffield), a large steel casting and forging supplier in the U.K., forged² the vessels in 2002 and 2003. Sheffield constructed the vessels from alloy steel to meet the material specifications of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code SA-723 Grade 2 steel³ (Appendix A). The 140,000-pound vessels have a maximum allowable working pressure (MAWP) of about 30,000 pounds per square inch gauge (psig) and maximum operating temperature of 750°F. Operators replaced the pressure relief device after each crystal growing cycle because of the high operating pressure. Each vessel had about three crystal-growing cycles per year.

1.2.1.1 Illinois State Special Vessels

After fabrication, Sheffield could not certify that the first three manufactured vessels were compliant with the ASME Boiler and Pressure Vessel Code (BPVC) because the vessel material did not meet the required mechanical properties for that class of steel. NDK petitioned the Illinois Boiler and Pressure Vessel Safety Division to grant special authorization to use the vessels. At a meeting held by the division in June 2002, technical representatives for NDK claimed that although the Charpy⁴ tests did not meet all the ASME Code requirements at lower temperatures, they showed acceptable material toughness at the operating temperature of 700°F. As an alternative to meeting code requirements, Sheffield asserted that the vessel material toughness was adequate and that the material passed the Charpy test at 212°F. The State had an independent party review the vessel design package. In September 2003, the State of Illinois gave NDK approval to use vessels 1, 2 and 3 and classified them as “state special” pressure vessels. Sheffield manufactured vessels 4 through 8 from a material with different mechanical properties that passed the Charpy impact test at 70°F.⁵ The manufacturer applied the ASME BPVC Section VIII, Division 3 Code stamp to those vessels.

FIGURE 2

A basket of mined quartz (lasca) prior to a crystal-growing run.



2.0 PROCESS DISCUSSION

2.1 PROCESS DESCRIPTION

The NDK quartz-manufacturing vessel simulated natural geologic crystal growth through heat and high pressure, known as hydrothermal synthesis. The process began with raw mined quartz, or lasca (Figure 2), lowered in baskets into the bottom of the vessel using an overhead crane. Operators then added 800 gallons of four percent sodium hydroxide and water solution, and a small amount of lithium nitrate into the vessels. Next, operators hung

² Forging is the shaping of metal between dies by mechanical or hydraulic pressure.

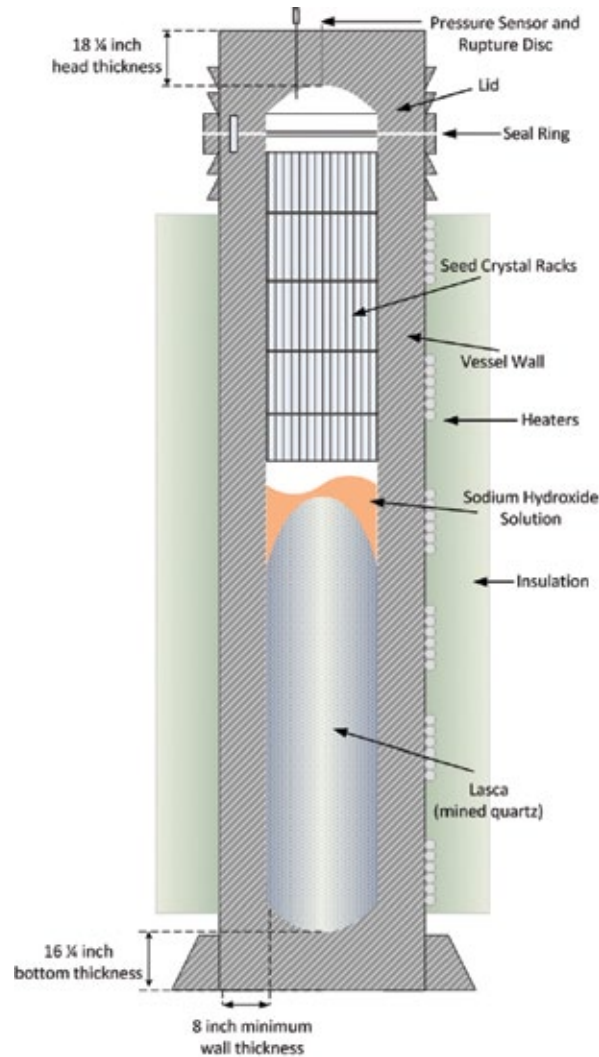
³ American Society of Mechanical Engineers (ASME) 2001, Specification for Alloy Steel Forgings for High-Strength Pressure Component Application, SA-723/SA-723M-2001. *ASME Boiler and Pressure Vessel Code Section II Part A*, ASME New York, NY.

⁴ The Charpy test is an impact test that measures the amount of energy a material can absorb.

⁵ Sheffield constructed vessels 1, 2 and 3 from alloy steel (SA-723 Class 3 Grade 2). Vessels 4 through 8 were forged from SA-723 Class 3 Grade 3 steel.

FIGURE 3

NDK Autoclave Cross Section (not to scale)



“seed crystals” at the top of the vessel. The seed crystals are wafer-thin pure quartz crystals with the desired grain structure on which the new crystals grow. Operators sealed the vessel with 10,000-pound lids clamped to the top. External electric heaters slowly increased vessel temperature to 700°F, which boiled the caustic liquid and increased the pressure inside the vessel to about 29,000 psig (Figure 3).

The natural quartz crystal, or lasca, dissolved in the liquid solution. Natural circulation caused the supersaturated solutions to rise to the top of the vessel, where the temperature was a few degrees lower. High purity quartz crystals grew on the seed crystals over a period of 100 to 150 days and formed a high-purity synthetic quartz product.

When the vessel run was completed and returned to ambient temperature, operators removed the finished quartz product and the racks from the vessel. NDK laboratory technicians examined the crystals for quality control purposes and shipped the product to the NDK facility in Japan for further processing (Figure 4).

Operators then cleaned the empty vessel interior with a pressure washer and vacuumed the water out of the vessel. All excess caustic and water solution was transferred to a holding tank where it was treated and neutralized prior to disposal.

In addition to NDK, there is one other crystal manufacturer in the U.S., located in Eastlake, Ohio. This company, in operation for 50 years, uses a sodium carbonate process and operates smaller, thinner-walled vessels at lower temperature and pressure than the sodium hydroxide process operated by NDK in Belvidere.⁶

FIGURE 4

Synthetic quartz product.



⁶ The sodium carbonate process uses 0.6 to 0.8M sodium carbonate solution. The vessels are heated to 650°F and operate at a pressure of 10,000 to 14,500 psi.

2.2 ACMITE COATING

The caustic sodium hydroxide solution and silica reacted with the iron in the steel vessel wall and formed a coating known as acmite.⁷ The presence of acmite, or sodium iron silicate, is significant in the manufacturing of synthetic quartz and its use has been well documented in lower-strength steels as early as 1954.⁸ Acmite serves a dual purpose for crystal manufacturing: to protect the surface of the vessel from corrosion and to avoid iron contamination in the final product. When NDK first received the vessels from the manufacturer, operators performed a “coating run” to apply a protective boundary between the liquid and the wall of the steel vessel.⁹

During the vessel operation, the acmite coating formed rapidly on the inner surface and coated the vessel wall. Because this high-strength, alloy steel vessel material was susceptible to corrosion, NDK relied on the acmite layer to prevent the alkaline process environment from corroding and weakening the vessel wall.

3.0 INCIDENT DESCRIPTION

On December 7, 2009, at approximately 2:30 pm, State Special Vessel No. 2, under an operating pressure of 29,000 psig, suddenly and violently ruptured, 120 days into a 150-day operating cycle. A white cloud of steam and debris rapidly expanded outward from the facility, traveled onto the interstate, and dissipated within seconds (Figure 5).

FIGURE 5

Oasis security camera shows the expanding steam cloud and debris immediately after the vessel ruptured.



The sudden release of superheated liquid caused an eight-foot tall by four-foot wide vessel fragment, weighing approximately 8,600 pounds, to travel through two concrete walls and finally land about 435 feet from the NDK building (Figure 6). The fragment skipped across a neighboring facility parking lot and slammed into the wall of an adjacent business office (Figure 7). The force of the impact pushed the wall inward causing furniture to shift and ceiling tiles to fall. One person working near the wall was injured.

⁷ Acmite, also known as aegirine, is a greenish-grey rock-forming mineral that is stable at high pressures and temperatures.

⁸ Walker, A.C., Ballman, A.A., “Synthesis of Quartz Crystal.” Bell Telephone Laboratories, 1954.

⁹ During the coating run, operators added lasca, seed crystal, the caustic solution, and a small amount of lithium nitrate to the vessel and heated the vessel to 650°F and 17,000 psig for 2 to 3 weeks.

FIGURE 6 (LEFT)

The 8,600 pound vessel fragment exited the NDK production building through a reinforced concrete wall.

**FIGURE 7 (RIGHT)**

Damage from vessel fragment impact with roadway.



The thrust from the escaping liquid caused the base of the vessel to violently shear away from its foundation and blew pieces of structural steel out of the building into the parking lot of a nearby rest stop gas station, known as the Illinois Tollway (I-90) Oasis. One piece of structural steel struck and killed a truck driver at the rest stop. After shearing from its base and throwing shrapnel out of the facility, the vessel swung from the building and landed on the ground outside (Figure 8).

FIGURE 8

NDK Crystal damage after the incident; Vessel No. 2 rests on ground outside structure. The force of the explosion blew out most of the exterior wall panels.



FIGURE 9

Structural steel fragment



FIGURE 10

Grupo Antolin facility damage from vessel fragment.



3.1 CONSEQUENCES

3.1.1 Fatality and Injury

At the time of the incident, a member of the public was leaving the I-90 Tollway Oasis and returning to his truck when he was struck by a flying fragment propelled by the force of the vessel explosion. The seven-foot, 100-pound steel beam (Figure 9) from the NDK facility traveled about 650 feet, and struck and killed the truck driver. An employee at Grupo Antolin, an adjacent automotive supply company, received minor injuries because of the impact from the 8,600-pound vessel fragment against the exterior wall of the office area. Emergency medical services evaluated and treated the Grupo Antolin employee on-scene. Six NDK Crystal employees were present in the production area at the time of the explosion, but were uninjured.

3.1.2 Property Damage

The NDK production facility sustained major damage. Exterior insulating panels were completely blown off. The force of the explosion and the displaced vessel destroyed steel framing, stairwells and floor grating near Vessel No. 2. The office and lab areas showed varying degrees of damage and much of the final product was destroyed. The product storage area, laboratory, and production offices were heavily damaged. The NDK America facility attached to the NDK Crystals production facility also received major damage from the vessel rupture. NDK America employees relocated office activities for three months during repairs.

The neighboring facility, Grupo Antolin, received damage to one wall, multiple ceiling tiles, and lights from the impact of the 8,600-pound projectile from the pressure vessel (Figure 10). Several automobiles in the parking lot sustained damage from the force of the explosion and flying debris. One piece of shrapnel, a 2-foot by 2-foot window, landed in the Oasis parking lot, 950 feet away (Figure 11).

FIGURE 11

Overhead photo of explosion fragment locations and distance from the facility.



3.2 EMERGENCY RESPONSE

The Belvidere Fire Department arrived on scene at 2:41 pm. The Chief of the Belvidere Fire Department assumed the role of incident commander. Employees evacuated the buildings and NDK management and the fire department accounted for all personnel. Fire department personnel escorted employees and bystanders involved in the explosion to the northwest parking lot of the Grupo Antolin facility, where the Belvidere Fire Department and Lifeline Ambulance set up triage. Responders also checked the immediate area using a thermal imaging camera and found the 8,600-pound portion of a vessel registering a temperature of 325°F. There was no sustained fire or smoke near the location of the explosion. Lifeline Helicopter airlifted the member of the public who was struck by the 100-pound steel beam to the hospital. He was pronounced dead at 3:15 pm that day.

The CSB learned of the incident through media reports and notification from the National Response Center. Because of the public fatality, the CSB deployed a team of three investigators who arrived on scene two days later. The CSB entered an evidence preservation agreement with OSHA, EPA and NDK Crystal, interviewed employees, photo-documented the site, and preserved physical evidence for further examination. NDK immediately shutdown all operations at the facility and on January 29, 2010, the Illinois State Fire Marshal's Office suspended operational certifications for all eight vessels at NDK.¹⁰

As of the publication date of this case study, the NDK Crystal facility remains shutdown. The seven remaining vessels are intact and contractors have performed significant demolition work to the surrounding superstructure.

4.0 ANALYSIS

4.1 MECHANISMS OF FAILURE

CSB investigators reviewed process data for the vessel over the 120 days prior to the incident and found no evidence of a process deviation that might have caused the vessel to fail.¹¹ As a result, the CSB entered into a joint metallurgical examination and testing agreement with OSHA, NDK, the insurance company, and other interested parties, to determine what caused the vessel to fail and whether the failure was caused by characteristics of the crystal-growing process or a problem with the vessel originating from its design and fabrication.

CSB investigators observed non-destructive evaluations (NDEs)¹² and destructive testing performed on the 8,600-pound vessel fragment (Figures 12, 13). After consultation with engineers and metallurgists from the various agencies, the metallurgical lab cut several samples from the vessel fragment inner diameter, outer diameter, thickness, and fracture surfaces for microscopic examination and testing. The metallurgical labs involved in the vessel fragment examinations provided all participating agencies with test data and photographs collected during testing. The CSB commissioned an independent review of the data by expert metallurgists at the National Institute of Standards and Technology (NIST) Materials Reliability Division, to assist in identifying the failure mechanism.

¹⁰ The vessel certifications remain suspended as of the publication date of this case study.

¹¹ The rupture disk on the lid of vessel 2 did not activate, indicating there was no excessive pressure build-up in the vessel prior to rupture. Vessel 2 pressure and temperature readings remained constant until the vessel ruptured.

¹² Non-destructive testing (NDT) is a wide group of surface and volumetric examination techniques used to evaluate the properties of a material, without causing damage. Common NDT methods include ultrasonic, magnetic-particle, liquid penetrant, radiographic, and visual inspection.

FIGURE 12 (LEFT)

8,600-pound vessel fragment subjected to metallurgical tests.

**FIGURE 13 (RIGHT)**

Photo of the ruptured Vessel No. 2 with insulation removed.

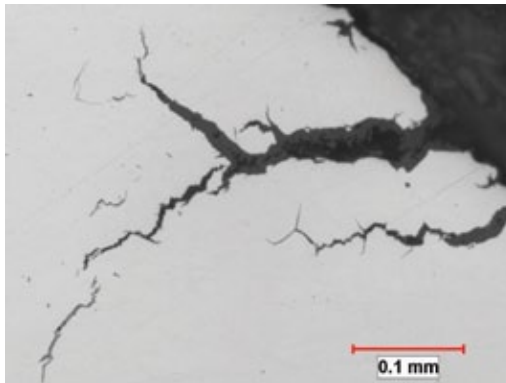


4.1.1 Stress-Corrosion Cracking

After a review of the metallurgical testing data, the CSB found strong evidence of cracking on and near the inner diameter of the vessel fragment. The cracks reduced the vessel material toughness,¹³ which eventually led to large flaws resulting in the catastrophic failure. Stress corrosion cracking (SCC) was the likely failure mechanism that caused the cracks. SCC is the formation of cracks through the simultaneous action of applied stresses and a corrosive environment.¹⁴ Microscopic examinations of the steel revealed strong evidence that SCC existed in many regions of the vessel fragment (Figure 14). The vessel at NDK contained an alkaline, or basic, sodium hydroxide and water solution generally known to damage some steels. The caustic liquid created a degrading environment inside the vessel, particularly in small surface scratches, and likely resulted in the development of SCC. The fracture initiated at an existing, surface-breaking crack in the inner diameter of the lower portion of the vessel near the base.

FIGURE 14

Micrograph of cracking found on the fracture surface about 0.75 inches from inner diameter surface of vessel fragment. According to NIST, the branching of the crack is a telltale sign of SCC.¹⁵



EPSI designed the vessels using the material NDK selected under the implied assumption that the acmite layer formation would prevent SCC in the vessel wall. The evidence of SCC in the fragment suggests that the acmite did not adequately protect the vessel, or that it was removed from this region of the vessel through some type of mechanical process, such as scratching or abrasion, during product removal or vessel cleaning between runs.

During the fragment testing, metallurgists performed energy dispersive spectroscopy¹⁶ (EDS) to characterize the coating on the vessel inner surface. Test results reported the presence of silicon, titanium, and aluminum in significant quantities, and sulfur and chloride in smaller quantities. These are known impurities for the lasca used as a feedstock for crystal growth. This evidence suggests that the process fluid was able to penetrate the surface cracks in the vessel, indicating the acmite coating was not adequate.

¹³ Toughness is the ability of a metal to resist fracture by absorbing the energy of applied stresses.

¹⁴ NACE International (2010). Stress Corrosion Cracking, NACE Resource Center. Retrieved from: <http://events.nace.org/library/corrosion/Forms/scc.asp>.

¹⁵ National Institute of Standards and Technology (NIST) Materials Reliability Division. (2013). Data Review and Report on Failure Mechanisms of NDK Pressure Vessel. Boulder, CO: Fekete, J. R., Sowards, J. W. & Amaro, R.

¹⁶ EDS is an analytical technique characterizes chemical elements on a sample surface.

4.1.2 Temper Embrittlement

Temper embrittlement is another mechanism of failure that may have contributed to the formation of the critical crack, or accelerated the SCC. Temper embrittlement is a phenomenon inherently present in heat-treated steels. Heat treatment is a process to alter the hardness and ductility throughout a material's microstructure. The control of time and temperature are critical to ensure a material has optimum mechanical properties for its intended use. Tempering is a heat treatment technique to decrease the steel hardness and increase ductility (decreasing brittleness), allowing the material to absorb more energy prior to failure.

4.1.2.1 Charpy Tests

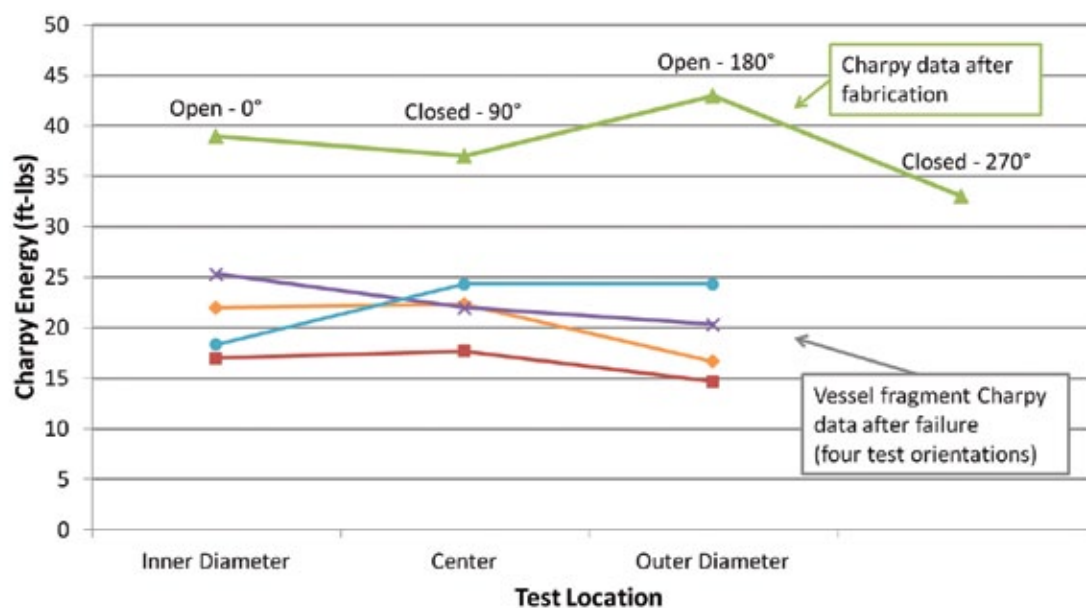
Metallurgists found possible evidence of temper embrittlement in the vessel sample from a review of Charpy impact test data. The Charpy impact test, also known as the Charpy V-notch test, is a standardized test, which determines the amount of energy absorbed by a material during fracture. A hammer on a pendulum arm strikes the sample specimen opposite the pre-machined notch. The energy absorbed by the specimen is determined by measuring the decrease in motion of the pendulum arm. This absorbed energy is a measure of a given material's notch toughness and along with the appearance of the fracture surface can determine the minimum service temperature of a material.

The CSB made comparisons between the manufacturing certification and post-failure testing, and found there were considerable variations in toughness in different regions of the vessel. This general loss of toughness was identified in all orientations (not just circumferential), which, according to NIST, suggests a general damage mechanism, such as temper embrittlement was acting in addition to the SCC (Figure 14). The reduction in Charpy energy shows that the vessel's ability to absorb energy before failure diminished over time, possibly caused by the SCC.

TABLE 1

Comparison of Charpy impact test results after manufacture and post failure. Manufacturer's Charpy data were taken from a different orientation. Post failure data points are the average of three tests in four orientations. All tests measured at 212°F.

VESSEL NO. 2 CHARPY ENERGY DECREASE POST FAILURE



4.1.3 Forging Process and Heat Treatment

The vessels at the NDK Crystal facility were all forged from a solid bar into a long cylinder, leaving the base of the vessel with an elliptical or “dished” end inside (Figure 3). The vessel lids came from the same ingot.¹⁷ The vessel was partially machined into shape, forged, heat-treated, and quenched. Quenching with water puts the steel in a harder or more brittle state, and then it is tempered back to reduce hardness and increase ductility.

The ASME Boiler and Pressure Vessel Code for this particular alloy steel¹⁸ has a 7-inch thickness limitation for quenching a closed-end vessel (Section 4.3.1.1). The NDK vessel maximum thickness at the top and bottom was more than double the “typical value” listed in the code. To determine if heat treatment issues contributed to the vessel failure, NIST reviewed the grain structure throughout the entire thickness of the vessel fragment. NIST concluded variations in grain structure through the vessel thickness were typical and there was no evidence that the vessel thickness resulted in inadequate heat treatment of the portion examined. The review of vessel fragment micrographs revealed expected microstructures for this type of steel.

Though NIST could not find evidence of inadequate heat treatment in the vessel fragment, it is possible that a reduction in toughness could have existed in the thicker regions of the vessel. Because of the thickness of the vessel at the top and bottom, Sheffield performed a double water quench in attempt to achieve the desired toughness in the center regions of the vessel wall. Early in the fabrication process, two vessels did not cool evenly during the quench process and cracked near the bottom. As a result, Sheffield discarded the vessels and requested NDK change the material specifications on the vessels yet to be forged, to Class 3 Grade 3 steel, with a higher nickel content to meet the toughness properties required by the ASME code.

4.2 NDK

4.2.1 Previous Incident

In January 2007, the closure head in Vessel No. 6 experienced an uncontrolled leak of the 400°F caustic sodium hydroxide solution that expelled through the threaded pressure sensor connection at the top of the vessel while in service. Hot, caustic material sprayed on the ceiling and fifth floor of the NDK Crystal facility. No one was injured as a result of the incident. NDK ceased production and initiated an investigation and examination of the vessel lids. A consultant hired by NDK conducted non-destructive examinations on the vessel lids and found very small cracks in four of the closure heads (Vessels No. 1, 4, 6, and 8) and noted the possibility of cracks in the lid of Vessel No. 2. The cracks were found near the machined surface of the transducer hole in the vessel lid. The consultant determined that the leak was caused by stress corrosion cracking and was present in four of the vessel heads. The consultant attributed the stress corrosion cracking to issues in the design, material selection, and heat treatment of the vessels and vessel lids.

4.2.1.1 Operating Warnings Prior to Incident

As a result of the vessel lid failure investigation, in July 2007 the consultant hired by NDK’s insurance company notified the insurer it had “serious reservations” about returning any of the vessels to service, specifically Vessels No. 3 and 5, and a decision to do so would be a “seriously flawed decision and not based on sound engineering analyses.” The consultant also cautioned that “far more catastrophic scenarios” involving the vessels were possible,

¹⁷ An ingot is a mass or block of metal before casting or forging.

¹⁸ American Society of Mechanical Engineers (ASME). 2001, Specification for Alloy Steel Forgings for High-Strength Pressure Component Application, SA-723/SA-723M-2001. *ASME Boiler and Pressure Vessel Code Section II Part A*. New York, NY.

imposing a hazard on NDK and the public, “including anyone who might be at the Belvidere Oasis on the Northwest Tollroad.” As a result, the insurer notified NDK of the consultant’s findings and reserved the right to deny claims for any future damages or consequences from the vessels (Appendix B). Despite this communication, NDK continued to operate the seven remaining vessels with intact lids, without performing thorough inspections of the vessel bodies by a certified professional. As of the publication date of this case study, the insurance company has not paid claims for the losses incurred by the 2009 explosion.

Even though the exact mechanism of failure for the vessel lids may have been different from the December 2009 rupture, the January 2007 incident and the results of the metallurgical examination should have prompted a closer examination of the vessel interior. The SCC on the lids was the first indication that the protective capability of the acmite coating was less than adequate, possibly throughout the entire vessel. Because the forging supplier produced the lids from the same ingot as the vessels, it was possible that a similar failure mechanism was acting on the vessel interior. The consultants investigating the lid failure indicated that the inner diameter was susceptible to cracks due to higher tensile hoop stresses down the length of the cylinder where caustic could concentrate in a surface notch or crack. The metallurgists commissioned by the CSB found NDK could have easily detected cracks in the vessel sample by non-destructive test methods, had the company performed such examinations. CSB investigators reviewed documents obtained by the insurance company and learned NDK was in the process of getting cost estimates for vessel body replacements at the time of the 2009 explosion.

4.2.2 Inspections

When NDK petitioned the State Boiler and Pressure Vessel Safety Division to get authorization to operate Vessels No. 1 through 3, the vessel designer from EPSI recommended annual, non-destructive inspections of the vessel interior. NDK has no documentation of any internal inspections of Vessel No. 2 throughout its six-year service life. Other than regular visual inspections of the top portion of the vessel between runs, NDK did not have a scheduled inspection program for entire length of the vessel interior.

After the 2007 lid investigation, the consultant hired by the insurance company recommended NDK perform inspections on the vessels prior to continuing operations. The consultant recommended a thorough interior examination of the vessels to identify cracks, as well as a Fitness-for-Service evaluation under the ASME and American Petroleum Institute (API) Recommended Practice 579.¹⁹ NDK contracted a metallurgical consultant to perform an ultrasonic inspection only on the top portion of Vessel No. 1 and found no internal discontinuities. Despite these warnings and the recommendation to inspect all vessels, NDK continued operation while focusing on the redesign of the lids without examining the origin of the SCC identified by the consultant. NDK performed weld repairs on the cracks near the pressure sensor connections on the vessel closure heads. The ASME code strictly prohibits welding for SA-723 forged steel.

In addition to the vessel inspections, there were several recommendations submitted by the insurer in 2008 that remained unfulfilled at the time of the incident. NDK’s approach to safety was informal, lacking formalized job training, standard operating procedures, and an incident and injury notification and investigation program.

¹⁹ Fitness for Service (FFS) assessment using API Recommended Practice 579 is performed to assess the operational safety and reliability process plant equipment.

4.3 CODES AND STANDARDS

4.3.1 ASME

The American Society of Mechanical Engineers (ASME) is a not-for-profit professional organization that develops codes and standards that cover many technical areas in mechanical engineering practice and provides professional development programs. The ASME Boiler and Pressure Vessel Code (BPVC) provides requirements for the design, fabrication, inspection, and certification of boilers and pressure vessels. The State of Illinois adopted the ASME BPVC in 1976.

4.3.1.1 Material Specifications

The BPVC Section II, Part A includes ferrous material specifications for pressure-retaining components. Within Part A, ASTM Standard SA-723, *Standard Specification for Alloy Steel Forgings for High-Strength Pressure Component Application*, covers the requirements for materials used for forging and heat-treated high-strength vessels like the crystal-growing vessels at NDK. It includes the chemical requirements for three primary classes of steel with six sub-classes based on increasing tensile strength (Appendix A).

The metallurgical examination commissioned by the CSB verified, through a chemical analysis of the sample, the material in Vessel No. 2 conformed to the chemical specifications as required by ASME. The three state special vessels at NDK could not meet the Charpy toughness levels required by ASME at 70°F, but did pass the Charpy tests at 212°F.

NDK and the vessel designer selected a vessel thickness that exceeded the typical thickness limit specified in the ASME Code. Table 2 and 3 of SA-723 state the “typical maximum section size” is 7 inches for closed-end vessels. The thickness limitation exists to ensure proper heat treatment is achieved. The NDK vessel cylindrical shell is 8 inches thick, and the top and bottom portions of the vessel had a thickness of 18 ¼ inches at the lid and 16 ¼ inches at the base. An analysis of the 8-inch thick Vessel No. 2 fragment microstructure did not show any evidence of a heat treatment issue; however, the 2007 examination of the 18-inch thick vessel lids revealed large grain sizes, indicating faulty heat treatments may have been present in the thicker sections of the vessels, such as the lids and the base.

The BPVC Section VIII Division 3, *Alternate Rules for Construction of High Pressure Vessels*, applies to vessels operating at pressures greater than 10,000 psig, such as the vessels at NDK. This section of the BPVC does not include specific limitations of material thickness for the design of heavy pressure vessels made with carbon and low-alloy steels, such as the Grade 2 steel used at NDK. Article KM-400,²⁰ Material Design Data, includes a table of required mechanical properties for each material covered under the section. The material listing used for the state special vessels has no required thickness limit.

4.3.1.2 Requirements for Corrosion Prevention

The design package for the NDK vessels did not mention the impact of the caustic service fluid on the interior of the vessel and the designer did not specify appropriate materials or other considerations, such as the acmite coating, for corrosion prevention. Article KG 311.7, Material Selection, in Section VIII Division 3 of the 2001 ASME code used for the design of the NDK vessels, required “appropriate materials for resistance to process corrosion including environmentally induced corrosion cracking.”²¹ In the user design

²⁰ ASME 2001, *Alternate Rules for Construction of Pressure Vessels, KCS-1 Carbon and Low Alloy Steels, UNS K34035. ASME Boiler and Pressure Vessel Code, Section VIII, Division 3*. New York, NY.

²¹ ASME 2001, *Alternate Rules for Construction of Pressure Vessels, Article KG 311.7, ASME Boiler and Pressure Vessel Code, Section VIII Division 3*. New York, NY.

package for the state special vessels, the documentation from the vessel designer states, “Designation of material by user for corrosion resistance: None specified” in response to the Section VIII Article KG 311.7 requirements for corrosion prevention.

In a subsequent issuance, ASME revised Section VIII of the code related to material design and selection of pressure-retaining materials. The BPVC Section VIII, Division 3, Article KM-400, Material Design Data,²² lists requirements for materials used in the fabrication of vessels, such as material limitations and mechanical properties. It contains additional requirements and cautionary statements listed for the Grade 2 Class 3 material used at NDK:

- (17) *Caution is advised when using the material above 700 deg F. After exposure to temperatures above 700 deg F, this material may exhibit temper embrittlement and stress relaxation defects. The designer shall consider these effects and their influence on the vessel.*
- (21) *Caution is advised when using these materials, as they are more susceptible than lower strength materials to environmental stress corrosion cracking and or embrittlement due to hydrogen exposure... The designer shall consider these effects and their influence on the vessel.*
- (22) *These materials shall not be used for applications when the material, when loaded, is in contact with water or an aqueous solution*
 - a. *These materials are permitted if the material is protected from contact by water or an aqueous environment...*

Based on the changes to the ASME code, the appropriate code enforcing authority can prohibit the use of vessels similar to those at NDK with aqueous environments.

4.3.3 NACE International

NACE International (National Association of Corrosion Engineers)²³ is a global organization that offers training, certifications, industry standards, and publications related to corrosion prevention. NACE membership includes a broad range of industries, and NACE corrosion prevention standards are referenced or adopted by other codes and industry consensus standards such as API, ASTM, and pipeline safety regulations.

The NACE Standard, *Avoiding Caustic Stress Corrosion Cracking of Carbon Steel Refinery Equipment and Piping* (SP0403-2008), establishes guidelines to prevent SCC when using caustics, particularly aqueous sodium hydroxide. NACE has published guidance for handling sodium hydroxide since the mid-1960s. Though mostly used in refinery applications, the guidelines and precautions in this standard specifically address carbon steel components, but can also apply to the material used in the vessels at NDK. The increased design strength of the low-alloy steel vessels made them more susceptible to corrosion.

The NACE standard states, “Caustic concentrations greater than 5 weight percent in the aqueous phase can produce SCC in carbon steel. Caustic SCC sometimes occurs in services with lower bulk fluid concentrations, usually in areas where local concentration effects occur.” NDK put 4 percent caustic in the NDK vessel. The standard also states that carbon steel is not suitable for use at temperatures greater than 215°F unless a nickel alloy is used.²⁴

²² ASME 2007, Alternate Rules for Construction of Pressure Vessels, Article KG 311.7, *ASME Boiler and Pressure Vessel Code, Section VIII Division 3 -2008 Addenda*. New York, NY.

²³ Corrosion engineers in the pipeline industry originally founded NACE as the National Association of Corrosion Engineers in 1943.

²⁴ NACE International. *Avoiding Caustic Stress Corrosion Cracking of Carbon Steel Refinery Equipment and Piping*, Caustic Soda Service Chart SP0403. Houston, TX, 2008.

NDK relied on the acmite coating to act as an impervious barrier between the caustic environment and the inner surface of the vessel. According to the NACE Standard, organic or inorganic coatings may be applied but care should be taken to ensure the coating performs acceptably during process operations, shutdown, and cleaning. However, the acmite was a coating formed during the crystal growing process to improve product quality; it was not specifically applied to the vessel for corrosion prevention. NDK did not perform regular inspections of the vessel interior to ensure the acmite coating was providing adequate corrosion protection. After the 2007 lid failure, NDK did not further examine the origin of the SCC issues on the lid material prior to putting the vessels back into service. This 2007 lid incident was the first indication the acmite coating was not providing complete corrosion protection.

5.0 REGULATORY ANALYSIS

5.1 IOSHA

The Illinois Regional Office of the Occupational Safety and Health Administration (IOSHA) investigated the December 2009 incident and in May 2010 issued several serious and willful citations to NDK. Though NDK employees were not hurt during the incident, IOSHA concluded NDK employees were exposed to the potential hazards associated with the crystal growing process in the absence of documented safety training, hazard communication for process chemicals, and emergency and evacuation procedures.²⁵ IOSHA also found that NDK did not complete a workplace hazard assessment for personal protective equipment. IOSHA cited NDK serious and willful violations under Section 5(a)(1) General Duty Clause,²⁶ for not evaluating the NDK building for explosion hazards using recognized industry practices, and for choosing to return the vessels to service after the 2007 lid examination showed evidence of a material defect.

5.2 IEPA

Following the December 2009 explosion, the Illinois Environmental Protection Agency (IEPA) proceeded with enforcement action against NDK for possibly polluting the soil, air, and groundwater with the sodium hydroxide solution released from Vessel No. 2 and the containment tanks during the explosion, and with the synthetic quartz crystals that may have released from the ruptured vessel. The State Attorney General filed a complaint against NDK following the explosion for alleged violations of the Illinois Environmental Protection Act.²⁷ IEPA issued violations for a substantial endangerment to the environment and air pollution. NDK entered into a consent order with the IEPA to settle these claims and agreed to participate in several compliance activities approved by the IEPA. NDK contracted with an independent environmental consulting company to collect samples, conduct an analysis of those samples to estimate the identity and quantity of process chemicals released to the environment during the explosion, and submit the report to the IEPA.

The environmental consultants determined through sampling collection, testing, and analysis that accumulations of dust in and around the NDK facility following the incident consisted of building construction materials disturbed during the explosion. Information from the sample tests was insufficient to determine if silica from the lasca was released during the explosion.

²⁵ The NDK Crystal facility was not subject to the requirements of the Process Safety Management (PSM) Standard because the process did not contain significant quantities of listed hazardous materials or flammables.

²⁶ Under the OSH Act of 1970, Section 5(a)(1), known as the General Duty Clause, states: "Each employer shall furnish to each of his employees employment and a place of employment which are free from recognized hazards that are causing or are likely to cause death or serious physical harm to his employees."

²⁷ Illinois Compiled Statutes, Environmental Protection Act § 415 ILCS 5.

Soil surface pH measurements were slightly influenced by the release, but the consultant concluded that the materials released during the explosion would not cause adverse environmental or health exposure risks.

IEPA imposed a civil penalty of \$39,000 on NDK. The company also paid response costs totaling \$10,000 to the IEPA and Boone County. IEPA further required that NDK not resume operating the pressure vessels unless the IL State Fire Marshal's office certified the vessels as safe for operation. The Attorney General also required NDK to submit a copy of the CSB investigation report on the NDK explosion, following publication.

5.3 STATE BOILER AND PRESSURE VESSEL INSPECTOR

The Division of Boiler and Pressure Vessel Safety within the Office of the Illinois State Fire Marshal regulates the construction, installation, operation, inspection, and repair of boilers and pressure vessels in Illinois. Illinois has over 100,000 boilers and pressure vessels registered and inspected on a routine basis by authorized, commissioned inspectors. In 1976, the state enacted the Boiler and Pressure Vessel Safety Act to establish the requirements of the division.²⁸ The Boiler and Pressure Vessel Safety Act applies to steam boilers, hot water boilers, and pressure vessels and includes requirements for inspector competencies, vessel inspection intervals, and state special pressure vessels. Under the act, the Division of Boiler and Pressure Vessel Safety adopts the ASME code and the National Board Inspection Code (NBIC). The NBIC is an American National Standards Institute (ANSI) code for boiler and pressure vessel inspectors, which provides standards for the installation, inspection, and repair of boilers, pressure vessels, and pressure relief devices.²⁹ It is adopted by the State of Illinois and establishes the requirements for all boiler pressure vessel inspections and repairs in the state.

The Boiler and Pressure Vessel Safety Act requires that pressure vessels subject to internal corrosion receive a certificate of inspection every three years.³⁰ However, when the NDK vessels received their initial certification from the state, the Board of Boiler and Pressure Vessel Safety approved the vessels for non-corrosive service. As a result, the State Boiler and Pressure Vessel Inspector only inspected accessible external surfaces and pressure relief devices. The State Inspector conducted an external visual inspection of Vessel No. 2 in 2003 and 2006 and finally on September 16, 2009, less than three months before the incident. According to the Boiler and Pressure Vessel Safety Act, internal inspections are not required of pressure vessels containing materials that are known to be noncorrosive to the materials of construction, either from the chemical composition of the materials or from evidence that the materials are adequately treated with a corrosion inhibitor.³¹ The caustic sodium hydroxide solution used at NDK is known to be corrosive to carbon steel. However, the Board of Boiler and Pressure Vessel Safety did not thoroughly examine the impact of the service fluid on the vessel during the meeting when NDK petitioned the state to use the vessels, or during the independent review of the vessel design package. When NDK petitioned the board to grant "state special" authorization, the designer from EPSI recommended annual, internal inspections such as magnetic particle and ultrasonic

²⁸ Illinois Compiled Statutes, Boiler and Pressure Vessel Safety Act § 430 ILCS 75.

²⁹ The NBIC is developed by the National Board of Boiler and Pressure Vessels Inspectors (NBBI). NBBI is an organization composed of chief boiler and pressure vessel inspectors throughout North America. Its purpose is to promote safety and education through training, enforcement guidance, and the development of applicable standards.

³⁰ Illinois Compiled Statutes, Boiler and Pressure Vessel Safety Act § 430 ILCS 75(10)(a)(4).

³¹ Illinois Compiled Statutes, Boiler and Pressure Vessel Safety Act § 430 ILCS 75(10)(a)(5).

examinations to ensure corrosion was not occurring inside the vessels.³² Despite this recommendation, NDK did not perform such inspections and there was no oversight from the Illinois Chief Inspector to ensure NDK was inspecting the vessels as recommended.

5.4 THE CITY OF BELVIDERE

The land that NDK currently occupies was originally undeveloped agricultural land. The land was designated as residential until 1996, when the City of Belvidere granted a zoning district change of about 200 acres of land to light industrial, office, and research space for economic development (Figure 15).

FIGURE 15

1996 Light-Industrial Zoning location near NDK Crystal.



In 2001, NDK petitioned the City of Belvidere to permit the construction of its proposed office and manufacturing facility. After scheduled hearings with the Belvidere Planning and Zoning Commission, and Building Department, the City granted NDK a permit and zoned the proposed NDK location as “light industrial.” NDK purchased a lot in the light industrial district near the I-90 Tollway Oasis.

The City zoned the NDK lot and the surrounding area as light industrial in accordance with the 1994 Belvidere Zoning Code that was adopted by the City at the time. The objective of the 1994 Belvidere Zoning Code was to protect and promote public health, safety and general welfare for the citizens of the city, including the prevention of bodily harm to persons or property by fire, toxic fumes, explosions, or other hazards.³³

The 1994 Zoning Code defined light industrial districts as appropriate for “uses that will have a minimal detrimental effect on surrounding properties.”³⁴ Light industrial districts included manufacturing and production operations, but excluded any of the uses permitted only in

³² Proceedings from the IL State Fire Marshal Meeting, June 5, 2002, T20-3.

³³ 1994 Belvidere, Illinois, Code of Ordinances, § 150.001(h).

³⁴ 1994 Belvidere, Illinois, Code of Ordinances, § 150.0016(a).

the heavy industrial districts.³⁵ Though synthetic quartz manufacturing is not specifically listed in the heavy industrial uses, the temperatures and pressures involved in this operation are similar to the heavy industrial processes listed in the code, such as the production of coal, coke, and tar; gas manufacturing; petroleum refining processes; metal and metal ore refining; power and steam generation; and paint and rubber manufacturing.³⁶

Due to the height of the proposed NDK facility, the City required a special-use permit. According to the Belvidere code, when considering a special use permit, the planning commission shall take into consideration several criteria prior to approval, including the protection of public health and general welfare, and that the special use will not cause substantial injury to the value of proposed locations or other property in the neighborhood.³⁷

CSB reviewed records from the City of Belvidere Planning and Zoning Commission and Building Department. According to the City of Belvidere's summary of findings in the special-use permit documentation, there was no evidence that the design, location, or proposed operation affected public safety. However, the City of Belvidere required compliance with all other applicable codes and ordinances as a condition for permit approval. The CSB found no documentation that the zoning commission considered the offsite consequences associated with the proposed operation of eight high-pressure vessels at the facility. The installation and operation of the pressure vessels for the manufacturing process was not included in the application or any documentation submitted to the City during the permitting process.

6.0 KEY FINDINGS

1. Stress corrosion cracking likely caused the catastrophic rupture of a high-pressure crystal production vessel at NDK Crystal, Inc., fatally injuring a member of the public 650 feet away at a highway rest stop.
2. NDK relied upon the in-process formation of an acmite coating inside the production vessel to protect the low alloy, high-strength steel from caustic sodium hydroxide used in the manufacturing process. However, NDK did not verify the integrity or effectiveness of this coating, and the caustic chemicals promoted stress corrosion cracking that weakened the vessel.
3. The Illinois Board of Boiler and Pressure Vessel Safety did not conduct internal inspections of the NDK vessels as required under state regulations for pressure vessels subject to internal corrosion. Instead, the state says it relied on the company to perform internal inspections, but it did not verify whether these were actually occurring. The state conducted three certificate inspections of the vessel that failed in 2003, 2006, and 2009 (less than three months prior to the incident) but these inspections focused only on accessible external surfaces.
4. In 2007, NDK learned that stress corrosion cracking was occurring in four of eight pressure vessel lids at the facility. A consultant to NDK's insurance company warned NDK of "serious reservations" about returning the vessels to service after this discovery and specifically cited the possible danger to members of the public at the nearby rest stop in case of a catastrophic vessel failure.
5. Despite the insurance company warning, NDK did not perform recommended non-destructive examinations of all vessels prior to returning the vessels to service.
6. NDK did not perform annual internal inspections as recommended by the vessel designer when the vessels were initially constructed.

³⁵ 1994 Belvidere, Illinois, Code of Ordinances, § 150.0016(b).

³⁶ 1994 Belvidere, Illinois, Code of Ordinances, § 150.0017(b).

³⁷ 1994 Belvidere, Illinois, Code of Ordinances, § 150.0024.

7. Temper embrittlement, or some other form of heat treatment embrittlement, cannot be ruled out as a contributing factor in addition to the SCC. The vessels exceeded the ASME wall thickness recommendations for closed-end forgings, which may have resulted in improper heat treatment during the manufacturing process.
8. The ASME Boiler and Pressure Vessel Code does not have specific wall thickness limitations for pressure containing components.

7.0 RECOMMENDATIONS

7.1 AMERICAN SOCIETY OF MECHANICAL ENGINEERS (ASME)

2010-4-I-IL R1 Revise the ASME Boiler and Pressure Vessel Code to include specific material thickness limitations for the design of pressure-containing components to ensure proper heat treatment and avoid environmentally induced damage mechanisms. Clarify required vessel wall thickness limitations for SA-723 steel in the following code sections:

- a) ASME BPVC Section II, Part A, Material Requirements
- b) ASME BPVC Section VIII, Division III, Article KM-400, Material Design Data

7.2 NATIONAL BOARD OF BOILER AND PRESSURE VESSEL INSPECTORS (NBBI)

2010-4-I-IL R2 Communicate the findings of this Case Study to pressure vessel inspectors in all 50 states. At a minimum:

- a) Send the information (such as an article summarizing the investigation) to each member who is a pressure vessel inspector; and,
- b) Post a direct link to the NDK Case Study on the National Board's webpage.

7.3 OFFICE OF THE ILLINOIS STATE FIRE MARSHAL, BOILER AND PRESSURE VESSEL SAFETY DIVISION

2010-4-I-IL R3 Prohibit the use of the existing pressure vessels at the NDK facility for crystal growing operations.

2010-4-I-IL R4 Develop and implement state requirements and procedures to ensure the pressure vessel approval process accurately identifies vessels that may be subject to corrosion or similar deterioration mechanisms, and ensure regular inspections in accordance with these state requirements.

7.4 NDK CRYSTAL, INC.

2010-4-I-IL R5 For the design and operation of any new NDK Crystal facility using a hydrothermal or equivalent crystal growing process, ensure that the facility uses a process that is rigorously demonstrated to be inherently safer than the existing process (for example through using lower temperatures and pressures, and/or less corrosive conditions).

2010-4-I-IL R6 Implement a program to ensure the ongoing integrity of any coating used on the new process vessels. Employ an expert (e.g., a coatings expert certified by NACE International (National Association of Corrosion Engineers)) to design the program.

2010-4-I-IL R7	Implement an annual inspection and corrective action program to ensure vessels remain resistant to environmentally induced damage mechanisms based on the inspection guidelines set forth in the American Petroleum Institute (API) Standard 510, Pressure Vessel Inspection Code.
2010-4-I-IL R8	Commission a facility siting study of the NDK Crystal facility by an independent consultant. Identify all vessel failure scenarios prior to restarting the crystal growing process. The siting study should include the consequences, necessary preventive measures, and emergency planning and response programs relevant to each failure scenario for all surrounding points of concern. Provide a copy of this study to the City of Belvidere Building and Zoning Department and the Illinois Boiler and Pressure Vessel Safety Division.

APPENDIX A

TABLE 1 - CHEMICAL REQUIREMENTS

	PERCENT COMPOSITION		
	Grade 1	Grade 2	Grade 3
Carbon, max	0.35	0.40	0.40
Manganese, max	0.90	0.90	0.90
Phosphorus, max	0.02	0.02	0.02
Sulfur, max	0.02	0.02	0.02
Silicon, max	0.35	0.35	0.35
Nickel	1.5 to 2.25	2.3 to 3.3	3.3 to 4.5
Chromium	0.80 to 2.00	0.80 to 2.0	0.80 to 2.0
Molybdenum	0.20 to 0.40	0.30 to 0.50	0.40 to 0.80
Vanadium, max	0.20	0.20	0.20

TABLE 2 - TENSILE REQUIREMENTS

	Class 1	Class 2	Class 2a	Class 3 ^a	Class 4	Class 5
Tensile Strength, min, ksi	115	135	145	155	175	190
Yield Strength, 0.2% offset, min, psi	110	120	130	140	160	180
Elongation in 2 inches or 50mm, min, %	16	14	12.5	13	12	10
Reduction of area, min, %	50	45	43	40	35	30

A) Typical maximum section size of 10 in. for open-ended vessels, or 7 in. for blind-ended vessels

APPENDIX B

3851 Exchange Avenue
Aurora, Illinois 60504



Phone: (630) 851-4566
Fax: (630) 851-4970
esi-website.com

VIA E-MAIL and U. S. MAIL

July 31, 2007

St. Paul Travelers
P.O. Box 776
Ottawa, IL 61350

Re: NDK Crystal Autoclave Investigation
ESJ File: [REDACTED]

Dear Mr. [REDACTED]:

As you are aware, on July 23, 2007, Dr. Kent Johnson of our Aurora, IL office attended a joint inspection at the NDK facility in Belvidere, IL on behalf of St. Paul Travelers. At this meeting, [REDACTED] obtained confirmation that two of the eight autoclaves that are the focus of this investigation had been returned to service by NDK (vessels #3 and #5).

As you will recall, Engineering Systems Inc. (ESI) expressed serious reservations about returning any of these vessels to service when we were first informed that this was NDK's intent. Given that four, and possibly five, of the eight vessels have already cracked after very few service cycles, there is a very real possibility that vessels #3 and #5 will crack in the near future.

If these production vessels were simply containing an innocuous liquid at very low pressures and temperatures, and the only consequence of a leak, or even a rupture, were release of the liquid with no real potential for injuring employees or damaging the facility, we would most likely not have any objection to NDK's decision. However, the operating conditions at the NDK facility are far more hazardous.

During crystal growing operations at the NDK facility, the subject autoclaves are required to contain a caustic solution that develops an internal pressure of 29,000 psi when brought up to the operating temperature of 750°F. Under the least catastrophic scenario, it is possible that a small leak could develop at a bore hole crack in a top closure, which could then result in the release of a high-pressure jet of caustic solution.

*Providing Clear Answers
Through Insights and Multidisciplinary Excellence*

COLORADO	FLORIDA	ILLINOIS	LOUISIANA	MISSOURI	TEXAS
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In the meantime, we understand that NDK has or will return two of the eight autoclave vessels to service (vessels #3 and #5) and is considering returning a third autoclave vessel to service.

We are enclosing a letter from ESI, dated 7/31/07, which expresses "serious reservations" about the return of these vessels to service. As stated in its letter, ESI believes it has a professional duty to notify you of the potential dangers associated with returning the vessels to service and has requested that we forward its concerns to you.

Please be advised that by advising you of ESI's concerns regarding the safety of operating these vessels, neither ESI nor Nipponkoa assumes any duty or responsibility for the safe operation of these or any of the vessels, and they expressly disavow any liability or responsibility for damage or injury to any person or property which may result from the continued use of these or any of the vessels in their current condition.

Please also be advised that should any property insured by Nipponkoa sustain loss or damage as a result of returning these vessels to service in their current condition, Nipponkoa reserves the right to deny any claim for such loss or damage on the basis that the loss or damage did not result from a fortuitous event but, rather, arose from a known loss and/or a loss in progress.

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With respect to the current claim, please be further advised that by continuing with its investigation of the loss, including advising you of ESI's concerns with respect to the return of the vessels to service, Nipponkoa does not waive any of its rights under the policy and reserves all defenses it may have with respect to all issues pertaining to the subject loss, including whether it has any liability for the loss.

Sincerely,


Nipponkoa Insurance Company

Attachment: ESI Letter of 7/31/07

3851 Exchange Avenue
Aurora, Illinois 60504



Phone: (630) 851-4566
Fax: (630) 851-4870
esi-website.com

VIA E-MAIL and U. S. MAIL

July 31, 2007

St. Paul Travelers
P.O. Box 776
Ottawa, IL 61350

Re: NDK Crystal Autoclave Investigation
ESJ File: [REDACTED]

Dear Mr. [REDACTED]

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As you will recall, Engineering Systems Inc. (ESI) expressed serious reservations about returning any of these vessels to service when we were first informed that this was NDK's intent. Given that four, and possibly five, of the eight vessels have already cracked after very few service cycles, there is a very real possibility that vessels #3 and #5 will crack in the near future.

If these production vessels were simply containing an innocuous liquid at very low pressures and temperatures, and the only consequence of a leak, or even a rupture, were release of the liquid with no real potential for injuring employees or damaging the facility, we would most likely not have any objection to NDK's decision. However, the operating conditions at the NDK facility are far more hazardous.

During crystal growing operations at the NDK facility, the subject autoclaves are required to contain a caustic solution that develops an internal pressure of 29,000 psi when brought up to the operating temperature of 750°F. Under the least catastrophic scenario, it is possible that a small leak could develop at a bore hole crack in a top closure, which could then result in the release of a high-pressure jet of caustic solution.

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July 31, 2007
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Obviously this scenario is not too far fetched because it has already happened. The only thing that prevented the serious injury or death of one or more employees was luck: no one was in the way of the stream.

Unfortunately, far more catastrophic scenarios are possible. If a crack were to grow to a critical size in which the stress intensity at the crack tip exceeded the fracture toughness of the high strength steels used in the vessels, a catastrophic rupture could occur. Under the best case scenario, in which there is only fluid in the autoclave and no compressed gas, the catastrophic rupture would "only" take the form of a sudden, violent release of a tremendous amount of strain energy stored in the structure as a result of operating at 29,000 psi.

This could result in fragmentation and collapse of the vessel, with a simultaneous release of the fluids and solids in the vessel. However, with no compressed gas to propel any of the large fragments, the failure might be fairly "local," but it could still cause damage to nearby vessels and, possibly, serious injury or death to any employees in the immediate vicinity of the vessel at the time of failure.

Something far worse could occur if there is any compressed gas in the autoclave, either as a result of failure to completely fill the vessel with liquid or through some other mechanism. Compressed gas can store a tremendous amount of energy and, if released suddenly as it would be during brittle fracture of one of the autoclaves, it can cause truly catastrophic damage similar to that caused by explosions.

In the case of the autoclaves, depending upon the amount of compressed gas, this could result in the violent fragmentation of a vessel with the possibility of large fragments either impacting other vessels or being completely ejected from the facility. Obviously, this scenario could also be deadly.

Given these potentially disastrous consequences, it is our opinion that vessels #3 and #5 should not have been returned to service until the metallurgical investigation was completed. Questions that remain unanswered at this point in time include the following:

1. Is there an environmental component to the root cause of the observed cracking?
2. Is there any additional cracking that has initiated on the inner surfaces of the lower portions of these autoclaves?
3. Has there been any embrittlement, which would cause a reduction in the fracture toughness of the autoclave/top closure materials, either due to vessel fabrication or service related conditions?

Without a more complete understanding of the root cause of the observed cracking and an adequate assessment of the actual fracture toughness of the high-strength steels used in the vessels, an informed decision with respect to the structural integrity of autoclaves #3 and #5 cannot be made.


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[REDACTED]
July 31, 2007
Page 3

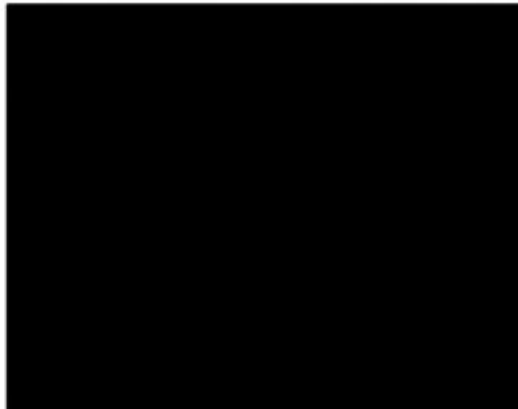
If NDK's recent decision to return these vessels to service was solely a management decision, and was not based on sound engineering analysis, comprehensive non-destructive flaw detection throughout the vessels, and finally, an API 579, Fitness-For-Service, Type III comprehensive evaluation, it is ESI's opinion that this decision was seriously flawed.

We are bringing this matter to your attention because, as professional engineers registered in the State of Illinois, we have an obligation to protect the welfare of the public. In this case, the "public" would include the employees of NDK Crystal, Inc., any visitors in or around the NDK facility, and anyone who might happen to be at the Belvidere Oasis on the Northwest Tollroad if one of these two vessels were to fail catastrophically. As noted above, a catastrophic failure of one or more vessels could result in the serious injury, or death, of any number of people in the surrounding area.

ESI hopes to be able to assist in the resolution of this problem, but would request confirmation that our concerns have been brought to the attention of the appropriate technical and management personnel at NDK and their insurers. Please provide us with written confirmation when this has been accomplished.



and




Engineering
Systems
Inc.
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CSB Investigation Reports are formal, detailed reports on significant chemical accidents and include key findings, root causes, and safety recommendations. CSB Hazard Investigations are broader studies of significant chemical hazards. CSB Safety Bulletins are short, general-interest publications that provide new or noteworthy information on preventing chemical accidents. CSB Case Studies are short reports on specific accidents and include a discussion of relevant prevention practices. All reports may contain include safety recommendations when appropriate. CSB Investigation Digests are plain-language summaries of Investigation Reports.

The U.S. Chemical Safety and Hazard Investigation Board (CSB) is an independent Federal agency whose mission is to ensure the safety of workers, the public, and the environment by investigating and preventing chemical incidents. The CSB is a scientific investigative organization; it is not an enforcement or regulatory body. Established by the Clean Air Act Amendments of 1990, the CSB is responsible for determining the root and contributing causes of accidents, issuing safety recommendations, studying chemical safety issues, and evaluating the effectiveness of other government agencies involved in chemical safety.

No part of the conclusions, findings, or recommendations of the CSB relating to any chemical accident may be admitted as evidence or used in any action or suit for damages. See 42 U.S.C. § 7412(r)(6)(G). The CSB makes public its actions and decisions through investigation reports, summary reports, safety bulletins, safety recommendations, case studies, incident digests, special technical publications, and statistical reviews. More information about the CSB is available at www.csb.gov.

CSB publications can be downloaded at www.csb.gov or obtained by contacting:
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