Hydrogen: Damage Mechanisms on Hydrogen Production Assets & Equipment
By Sean Berg, Ph.D., P.E., CFEI
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In previous articles in this series, BakerRisk discussed the hydrogen economy and high-level hazards. Additionally, best practices and general guidance for performing a PHA on hydrogen processing facilities to accurately assess all the risks and consequences associated with hydrogen were also covered. This article will focus on the materials and potential damage mechanisms and hazards associated with operation of equipment exposed to hydrogen, including pressure vessels, pipelines, piping, and other equipment handling hydrogen.

Equipment involved in hydrogen production, including electrolyzers, flow batteries, fuel cells, and ammonia chemical plants, typically operate at elevated temperatures and are exposed to environments where hydrogen partial pressures or concentrations can potentially result in life-limiting conditions for such equipment, which generally lead to loss of containment and possible catastrophic failure. Accommodating a growing usage of hydrogen for energy applications requires more production capacity over the next several years; as well as increased ammonia production capacity and more global ammonia facilities, in addition to boosting the volume of electrolyzers and other hydrogen technology.[1] Due to increased production and the exposure to hydrogen in said process equipment, there is potentially greater risk for hydrogen-related damaged to equipment and piping.

**Hydrogen, a Historic Perspective**
Awareness of hydrogen-related damage mechanisms that can damage equipment operating in hydrogen production applications is essential in developing the appropriate solution for proper equipment inspection, damage mitigation, and failure prevention. The pertinent damage mechanisms can also provide input for fitness-for-service evaluations, as the mechanism and rate of attack must be understood, to determine the remaining life of the equipment. For a proper risk-based inspection (RBI) program or during a hazards analysis, the appropriate hydrogen-damaged mechanisms should be identified so that the probability of failure can be determined when addressing reliability issues.[2]
Four hydrogen-related damage mechanisms affect hydrogen equipment.

- Hydrogen embrittlement (HE)
- Hydrogen induced cracking (HIC)
- Stress-oriented hydrogen-induced cracking (SOHIC)
- High temperature hydrogen attack (HTHA)

Hydrogen-related Damage Mechanisms

The following four hydrogen-related damage mechanisms have been observed to affect hydrogen equipment, and will be discussed in relation to process variables, process equipment, and materials of construction:

- Hydrogen embrittlement (HE)
- Hydrogen induced cracking (HIC)
- Stress-oriented hydrogen-induced cracking (SOHIC)
- High temperature hydrogen attack (HTHA)

This paper provides examples and discussion regarding each type of hydrogen damage mechanism relating to ammonia process parameters, materials of construction and process equipment, and inspection methods and characterization techniques to identify each form of damage. Additionally, useful tips for considering operating envelopes or limits and potential mitigation methods are provided.

Hydrogen Production Equipment Materials of Construction

Most equipment used in hydrogen production processes essentially consists of pressure vessels, piping, and storage tanks whose pressure boundaries are constructed of metallic materials. All materials of construction used in various hydrogen production industries are susceptible to degradation and various types of damage mechanisms due to the hydrogen. While it may be possible to select materials of construction that are completely resistant to attack by process fluids, sometimes such an approach can be impractical or cost prohibitive.

Carbon, low alloy, and stainless steels are the most commonly used materials of construction for process equipment. These materials offer a suitable combination of strength and ductility and are capable of safely operating in the temperature ranges typically employed. However, carbon and various alloy steels are susceptible to hydrogen damage mechanisms when exposed to a hydrogen environment. Hydrogen may take an atomic (H) or molecular hydrogen (recombined to H₂) form. In either case, only atomic hydrogen (H) diffuses into susceptible alloys such as ferritic steels (C-steel or Cr-Mo alloy steel). The hydrogen molecule (H₂) is too large to diffuse into these steels.
The use of stainless steels can reduce susceptibility to hydrogen damage mechanisms. However, austenitic stainless steel can also be susceptible if martensite has formed by heavy cold deformation. Titanium sometimes used in equipment has excellent resistance to general corrosion but can form hydrides if in a hydrogen environment.

**Comparison of Hydrogen-related Damage Mechanisms for Steel**

Hydrogen can be driven into metal by a corrosion reaction, pressure, partial pressure of hydrogen, temperature, concentration gradient, residual stress, or applied stress in the material. Hydrogen can be present in the metal through fabrication, such as in moist welding materials or in a humid welding environment. The microstructure of the material may also influence the location of where the damage may occur. The hardness of a material, weld or heat affected zone is an important factor. There is a hardness limit beyond which hydrogen damage may occur, often in lower temperature hydrogen damage.

Terminology describing hydrogen-related damage mechanisms can be confusing, inaccurate, or unclear. To identify similarities and help clear up the differences, the four common hydrogen damage mechanisms are described in Table 1 [3]. Table 1 provides an overview and comparison of each type of damage mechanism for carbon, low alloy, and stainless steels, including the form the hydrogen takes, description, key parameters, and key mitigation recommendations.[3]

API RP 571 provides general guidance as to the most likely damage mechanisms affecting common alloys used in the refining and petrochemical industry and is intended to introduce the concepts of service-induced deterioration and failure modes. However, as the API standards tend to focus on refining and hydrocarbon processes, the high pressure and temperature conditions in ammonia, methanol, and electrolyzer hydrogen production provides a wealth of experience and knowledge base for hydrogen damage mechanisms. This combined experience provides information that can be utilized by plant inspection personnel to assist in identifying likely causes of damage, to assist with the development of inspection strategies and to help identify monitoring programs that ensure equipment integrity.
**Table 1. Comparison of Hydrogen-Related Damage Mechanisms for Steel [3]**

<table>
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<th>Damage Mechanism</th>
<th>Hydrogen Form</th>
<th>Description</th>
<th>Key Parameters</th>
<th>Mitigation Strategies</th>
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| Hydrogen Embrittlement (HE)                   | Atomic        | Diffusion of hydrogen atoms into steel, which tend to migrate to voids and dislocations, applying pressure to interior of material and pinning dislocation motion. Results in brittle behavior. Typically occurs at temperatures below 150 °C (302 °F); following welding, plating, or submerged cathodic protection; when hydrogen is produced; or during elevated temperature service (>200 °C) (392°F) when hydrogen cannot diffuse out of steel and is then cooled (shutdown and subsequent startup). | • Strength and hardness  
• Residual stress  
• Material microstructure, e.g., heat affected zone  
• Atomic H-concentration in the steel after charging hydrogen during elevated temperature service. | • Limit steel hardness to <237 HB/HRC 22 or use lower strength steel  
• Hydrogen bake-out at 204 °C (400 °F) following weld processes  
• Allow outgassing of the steel during shutdown, i.e., lower the cooling rate and be aware of Minimum Pressurization Temperature curves. |
| Blistering and Hydrogen Induced Cracking (HIC)* | Molecular     | Exposure to hydrogen environment or wet H₂S (hydrogen produced during formation of FeS). Hydrogen enters in atomic form, but damage occurs after H-atoms recombine to H₂ molecules inside alloy. Blistering and HIC are strongly affected by the presence of inclusions, laminations (both found in “dirty” steels), and internal discontinuities, all of which provide sites for hydrogen accumulation. | • Hardness and strength  
• Residual stress  
• Temperature  
• Corrosion reaction with formation off atomic Hydrogen (such as H₂S)  
• Low impurities in the steel composition  
• Surface scales and inhibitors | • Limit steel hardness to below 237 HB/HRC 22  
• Use of protective lining in H₂S environment  
• Chemistry and manufacturing methods can affect susceptibility and can be modified to produce HIC resistant steel (refer to NACE 8X194). |
| Stress-Oriented Hydrogen Induced Cracking (SOHIC)* | Molecular     | Similar to HIC but cracking occurs in a sufficiently high stress field. Blisters or cracks stacked on top of one another and link up through the cross-sectional direction (e.g., stair-step cracks).                                                                                                                                  | • Same as HIC  
• Stress level applied to alloy                                                   | • Same as HIC  
• Lower stress level, if possible  
• Post-weld heat treatment |
| High Temperature Hydrogen Attack (HTHA)        | Atomic        | Diffusion of hydrogen into steel at elevated temperatures (>200 °C) (392 °F). Hydrogen reacts with carbides at elevated temperatures to form methane gas. Micro voids, grain boundary voids and micro fissures are generated. Buildup of methane pressure can result in blistering, degradation, and fissures within metal. | • Refer to most recent edition of Nelson Curves in API RP 941  
• Operating temperature  
• Partial pressure of hydrogen  
• Material selection                                                                  | • Monitor pressure, hydrogen partial pressure, and temperatures  
• Operate with a safety margin, e.g., 28 °C (50 °F), below the Nelson Curve  
• Post-weld heat treatment at sufficiently high temperatures that allow stable carbides to form  
• Use higher alloyed steels                                                            |

*Note: HIC and SOHIC are mainly related to feedstocks containing H₂S. They can occur in the gas cleaning section where H₂S is removed (i.e., before the “normal” ammonia plant equipment). After the desulfurization vessels, the amount of H₂S is generally too low to cause HIC or SOHIC.*
**Hydrogen Embrittlement**

Hydrogen embrittlement (HE) is the process by which various metals, most importantly high-strength steel, become brittle and fracture following exposure to hydrogen. Hydrogen embrittlement is often the result of unintentional introduction of hydrogen into susceptible metals during forming or finishing operations such as welding and plating.[4]

The mechanism starts with lone hydrogen atoms diffusing through the metal. At high temperatures, the elevated solubility of hydrogen allows hydrogen to diffuse more easily into the metal (or the hydrogen can diffuse in at a low temperature, assisted by a concentration gradient). The hydrogen atoms pin the dislocations in the steel, especially at the tips of cracks and internal defects. This results in limited moveability of the dislocations and brittle behavior of the steel. High-strength and low-alloy steels and nickel and titanium alloys are most susceptible. Also, Cr-Mo steels that have not been properly post-weld heat treated are susceptible. High hardness zones appear due to the formation of bainite or martensite.

During high temperature service the steel can be charged/loaded with hydrogen. When the process is taken out of service, hydrogen may get trapped inside the crystalline structure of the steel, especially for thick-walled components.

**Key Parameters and Factors:**

- Hydrogen must be present at a critical concentration within the steel and/or alloy.
- Increased risk where equipment temperatures are high, which can increase the solubility of hydrogen in the material.
- The strength level and microstructure of the steel/alloy must be susceptible to embrittlement. High strength steels above 237 HB/22 HRC are particularly sensitive to HE and can suffer delayed cracking before use due to the presence of hydrogen and residual stresses. Steel and welds with hardness of less than 237 HB/22 HRC are not generally considered susceptible to hydrogen embrittlement.
- A stress above the threshold for HE must be present from residual stresses and/or applied stresses.
- HE cracking can initiate sub-surface, but in most cases are surface-breaking.
- HE occurs at locations of high residual or tri-axial stresses (notches, restraint) and where the microstructure is conducive, such as in weld HAZs.
- In higher strength steels, cracking is often intergranular (may be transgranular) and can start subsurface.
- Welding – If wet electrodes or high moisture flux weld electrodes are used, hydrogen can be charged into the steel. Improper post weld heat treated (PWHT) welds or non-PWHT in pipes and vessels are susceptible.
- HE is most pronounced at temperatures between ambient to about 150 °C (300 °F) because the atomic hydrogen can diffuse at the elevated temperature (i.e., the hydrogen is mobile).
Mitigation Options:

- Use low hydrogen dry electrodes during welding and preheating methods.
- Bake electroplated steel components at temperatures of 190 to 220 °C (375 to 430 °F) within a few hours after the electroplating process.
- Use lower strength steels and reduce residual and applied stresses to avoid fracture due to hydrogen embrittlement.
- Apply proper post-weld heat treatment and reduce the hardness below 225-250 HV
- Allow outgassing of the hydrogen out of the steel by applying a proper (slow) cool-down procedure

Hydrogen Embrittlement Example [3]

Example: Titanium Hydride
Titanium can be very susceptible to titanium hydride formation. Titanium parts that absorb hydrogen at elevated temperatures can form hydrides upon cooling. These hydrides lead to a decrease in the strain to failure, loss of strength, and ductility. Hydrogen absorbed when present above specification can result in metal embrittlement, hardening, cracking, and spalling due to the formation of hydrides in the metal.

Background on the Equipment: A titanium 3Al-2.5V float was in an ammonia unit [5] The titanium float system is a magnetic level indicator that consists of a chamber and a magnet-equipped titanium float that raises and lowers with the fluid level. Operators reported problems with the float and erroneous level readings to the site supervision. The titanium float (shown in Figure 3) was removed for investigation and was found cracked, as shown in Figure 4.

Figure 1. The titanium float

Figure 2. Close-up of the weld showing cracks
The typical operating temperature was 133 °C (271 °F), the typical pressure was 2.8 MPa (400 psig), and the process medium included: Water 10.4%; Hydrogen 54.7%; Nitrogen 18.3%; and CO₂ 15.9%.

Findings: Metallurgical analysis determined that the cracking and spalling of the titanium float was due to the excessive pickup of hydrogen, which resulted in the formation of embrittling titanium hydrides.[5] The gas analysis of the weld region showed absorption of excessive amounts of hydrogen at the weld and heat affected zone. The weight percent of hydrogen was as much as 0.479% in the weld compared to 0.015 wt.% in the non-cracked sheet metal.

The most likely source of the hydrogen was from the operation in a hydrogen, steam, and ammonia environment. The areas where the most hydrogen was absorbed were at the weld and weld heat affected zones, likely due to residual stresses. These regions were also harder.

Figure 5 shows the affected weld and HAZ.

![Figure 3. Cross section through the weld and HAZ showing the cracking and hydrides](image)

Mitigation Options:

- Because the titanium absorbed hydrogen, action was taken to include a design change by applying a PTFE (Teflon-like) coating over the titanium float to prevent cracking from occurring. While the manufacturer provided a replacement titanium float with a PTFE coating, this coating may not be protective enough. Other more suitable metals could have been considered for replacement. It is best to keep titanium away from areas with high ammonia concentrations due to titanium’s affinity for nitrogen and hydrogen.

HIC and SOHIC

Hydrogen-induced cracking (HIC) and stress-oriented hydrogen induced cracking (SOHIC) are related. Both occur from the presence of hydrogen or from corrosion where hydrogen is liberated and diffuses into the steel, such as with wet H₂S corrosion. Hydrogen blisters may form as visible surface features or within the material. These blisters may be in arrays. HIC is a potential problem, mostly in low alloy steel weldments, and especially equipment fabricated in the 1960s that has high hardness and/or high levels of impurities and segregations.[6]

SOHIC occurs when these arrays of cracks are stacked on top of one another, usually in the base metal adjacent to the heat affected zone (HAZ).[7] This often occurs at high localized applied or residual stresses.
Key Parameters:

- HIC occurs at locations of high residual or tri-axial stresses (notches, restraint) and where the microstructure is conducive, such as in weld HAZs. Explosion cladding will generate high stresses.
- High hydrogen partial pressure
- Higher hardness alloys or local areas in welds are more susceptible to HIC and SOHIC
- Proper PWHT is required to ensure lower hardness is obtained
- Manual welds made with moisture in the electrodes
- Increasing H₂S potential and temperature increases the available hydrogen. Blistering has been observed between ambient and 150 °C (300 °F).

Mitigation Options:

- Carbon steels should be controlled to keep the weld hardness below 200 HB. Localized areas of hardness above 237 HB can be susceptible.
- Temper or PWHT procedures should be carefully followed and documented. Hardness testing with follow-up PWHT should be performed if hardness levels exceed recommendations.
- Some designers are moving away from Vanadium steels due to difficulty with fabrication.
- Where possible, conduct a furnace stress relief (instead of a local stress relief).
- If necessary, use alloy cladding to protect the steel from the H₂S corrosion.
- Use clean steels, like so-called HIC-resistant steels (NACE 8X194)

High Temperature Hydrogen Attack (HTHA)
High Temperature Hydrogen Attack (HTHA) is a form of degradation caused by hydrogen reacting with carbon to form methane in a high temperature environment.

\[ C + 4H \rightarrow CH_4 \]

When steel is exposed to hydrogen at elevated temperatures, hydrogen will diffuse into the alloy and react with carbon to form cavities/voids filled with methane.[8]

The methane is trapped in these voids and does not diffuse out of the metal. Over time, more and more methane is formed, forming more cavities at the grain boundaries. These cavities coalesce and form micro-cracks at the grain boundaries, which later will grow into macro-cracks. An example of such fractures can be seen in the microstructure of a pipe weld in Figure 6,[9] which shows a decarburization and fissuring region caused by hydrogen depleting the iron carbides. The cracks lower the rupture ductility and fracture toughness, which may result in brittle fracture. The brittle behavior of the material can result in a catastrophic brittle fracture of the asset [10-12] during startup or shutdown excursions.

Susceptible materials include plain carbon steels, C-½Mo steels, and other low alloy steels and non-PWHT welds. API RP 941 provides guidance to aid in materials selection for fixed equipment operating in environments with hydrogen partial pressures at elevated temperatures and pressures.[13]
This guidance can also be useful to materials engineers and process engineers alike, as knowledge of both process conditions and the materials of construction will provide information on an asset’s susceptibility to this particular damage mechanism.

The most obvious equipment concerns are any equipment exceeding normal operating temperatures or operating window limits, specifically carbon and low alloy steel vessels and piping operating at temperatures that are above the API 941 RP Nelson Curve values. These exceedances may not occur during normal operation, but may occur during startup, shutdown or upset conditions. Catalyst changes, fouling, and flow irregularities may also produce localized areas in exceedance of the recommended limits. Aging plants should be mindful of API RP 941 Nelson Curve operating point changes and should determine whether process changes or HTHA mitigation strategies may be implemented. HTHA is not a concern in solid stainless steel vessels. API RP 941 recommends not to take credit for the presence of a stainless steel cladding or weld overlay when selecting the base metal for a new vessel.

![Figure 4. Hydrogen damage observed in the carbon steel line at the heat affected zone (HAZ). Nital etch. (Original magnification: 200x).][3](image)

Hydrogen content is high in ammonia industry process streams, and so it is important to evaluate the potential for HTHA where the temperatures rise above 204 °C (400 °F) for carbon steel materials. The hydrogen content should be considered on a wet gas basis.

**Key Parameters:**

- Non-post weld heat treated steels are more susceptible.
- Operating temperatures within, e.g., 28 °C (50 °F) of the API RP 941 Nelson Curve values make the material more susceptible due to measurement capability.
- Environmental conditions: Hydrogen partial pressure and operating temperatures as susceptibility to attack increases as $H_2$ partial pressure increases.
- Materials with an inadequate safety factor using the API RP 941 Nelson Curve.
- Material substitutions with a wrong material or welding rods and equipment that are not inspected by Positive Material Identification (PMI).
• Material substitution can also happen during maintenance activities when, e.g., bends before and after heat exchangers are accidently mixed up (same dimensions, different steel)

• Exceeding normal operating temperatures or operating window limits.

• Refractory-lined vessels or refractory protected nozzles/pipes where refractory has been compromised (hot spots).

• Aging plants that have inadequate information on API RP 941 Nelson Curve changes, especially for C-½Mo steel and non-PWHT carbon steel.

• Stainless steel-lined vessels with the possibility of hydrogen entering behind the liner.

• Carbon steels and low alloy steels at operating temperatures that are above the API RP 941 Nelson Curve values, including processes that stray outside of the target integrity operating window (IOW).

**Equipment Concerns:**
Aging vessels in particular are subject to HTHA and other hydrogen mechanisms because of their years of service, potential operating excursions, and initial materials selection. In the ammonia industry, the methanator is a high temperature vessel that is subject to temperature excursions and is a candidate for HTHA along with the other methanation unit equipment. There have been reports of progressive degradation when HTHA is found in methanators, with the most notable damage on the bottom of the vessel, which experiences higher temperatures. HTHA damage should be monitored, and a Fitness for Service (FFS) evaluation considered to ensure integrity is maintained.

**Mitigation Options:**
One of the best ways to mitigate the potential for HTHA is for plant engineering to review plant processes, the design basis or Form U1 Manufacturer’s Report which includes the materials of construction, and operating conditions to identify potential HTHA risks with hydrogen-containing equipment. An important component of this includes conducting an engineering review of pressure, hydrogen partial pressure, and temperatures. Operating with safety margins, e.g., 28 °C (50 °F) below the API RP 941 Nelson Curve, can also provide additional assurance. Engineering should establish integrity operating limits for all vulnerable equipment. Having an active PMI and retro PMI program is also an essential mitigation component.

If possible or feasible, aging plants should consider replacing equipment with higher alloyed material that are less susceptible to HTHA according to the API RP 941 Nelson Curve for desired operating conditions. This review should include determining whether welded equipment or piping was post-weld heat treated, and if not known, assume non-post weld heat treated welds and operate at lower temperature and pressures. Another option is to consider performing PWHT during the next opportunity. Installing temperature indicators at critical locations, to monitor actual temperatures, and performing regular thermography measurements can help to ensure operating windows and limits are not exceeded or can be addressed.

• Review plant to identify potential HTHA risks with hydrogen-containing equipment.

• Perform regular thermography measurements.

• Have an active PMI and retro PMI program.
• Operate within safety margins, e.g., 28 °C (50 °F) below the API RP 941 Nelson Curve.
• Review quality of past repairs and ensure PWHT practices did not introduce an HTHA risk.
• Determine whether welded equipment or piping is post-weld heat treated. If not known, then assume non-post weld heat treated and operate at lower temperature and pressures. One may also consider performing PWHT during the next opportunity.
• Replace equipment with higher alloyed material that is less susceptible according to the API RP 941 Nelson Curve.
• Install temperature indicators at critical locations to monitor actual temperatures.
• Conduct engineering review of pressure, hydrogen partial pressure, and temperatures.
• Set up integrity operating limits for equipment.
• Recheck IOWs in conjunction with process changes where temperatures are affected, such as SynLoop converter retrofits for higher efficiencies.

HTHA Example [3]:
Background: An investigation conducted into a carbon steel effluent cooler header piping rupture, installed in an ammonia converter and synthesis loop, occurred 5 years after a change in operating conditions.[14] The process temperature was increased from 232 to 254 °C (450 to 490 °F), and the operating pressure was decreased from 29.0 MPa (4200 psig) (0.1 MPa (2100 psig) hydrogen partial pressure) to 23.4 MPa (3400 psig) (0.8 MPa (1700 psig) hydrogen partial pressure).

Findings: This process change placed the carbon steel pipe above the API RP 941 Nelson Curve temperature for carbon steel at the corresponding hydrogen partial pressure. The piping rupture was found to have a brittle fracture appearance. Failure analysis revealed that HTHA was the damage mechanism that caused the pipe rupture. This example case demonstrates the vulnerability of this portion of the ammonia process if material limits are exceeded and how process changes can create the potential for eventual failure.[14]

Mitigation options considered:
• Conducted Hydrogen damage review of equipment.
• Replaced piping with higher alloy material.

Inspection Methods:
For HE, HIC, and SOHIC, some common inspection methods such as those listed below can be used:
• Penetrant testing (PT)
• Magnetic particle testing (MT)
• Wet fluorescent magnetic particle testing (WFMT)
• Ultrasonic testing (UT)
• Radiographic testing (RT)
It must be noted that for volumetric (through wall thickness) HTHA inspections, the previous suggestions found in API RP 941 Annex E will be discontinued and replaced with API RP 586 (in balloting process at this time). It has been demonstrated that the historical methods have been supplanted by more advanced modalities of the Ultrasonic Testing methods found in API 586. These include Advanced Phase Array (using 64 Element Arrays), Total Focusing Method (TFM) or Full Matrix Capture (FMC), Time of Flight Diffraction or a combination of these methods. As always, these methods are strictly dependent on the technique and skill level of the inspector. There is a recognized training and certification program for these methods to qualify and maintain personnel competency.

Inspection methods that can identify potential regions of HTHA are:

- High Sensitivity Wet Fluorescent Magnetic Particle Testing (WFMT)
- Replication of surfaces
- Positive Material Identification (PMI)
- Thermographic temperature surveys

**Summary:**
Hydrogen-related damage mechanisms are present in hydrogen producing equipment. Knowledge of these hydrogen damage mechanisms and understanding how operations or process conditions can affect equipment can help personnel mitigate the occurrence of hydrogen-related damage mechanisms in their equipment. By making better distinctions among the three main hydrogen-related damage mechanisms, better identification and mitigation of the damage mechanism can be achieved.

Our next paper in this series will discuss facility siting studies for hydrogen applications, and will cover BakerRisk’s approach and methodology for effective hazard and threat identification through such studies.

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References:


