Assessing Aggregates for Radiation-Shielding Concrete

Methods for petrographic examination of high-density and boron-bearing aggregates

by Sang Y. Lee, Ann M. Daugherty, and Donald J. Broton

Radiation-shielding concrete (RSC) is used in nuclear power plants, health care facilities conducting radiation therapy, nuclear research facilities, and storage/transport casks for radioactive waste. In RSC mixtures, high-density aggregates are used to attenuate gamma rays and light atomic weight aggregates are used to absorb neutrons, so their properties and proportions will affect a mixture’s radiation-shielding characteristics.

The most common naturally occurring aggregates used in RSC are produced from ores of high-density minerals such as hematite, ilmenite, magnetite, and barite. Other aggregates used in RSC include aggregates that contain bound water, produced from ores of hydrous iron, serpentine, or bauxite, and aggregates that contain boron, produced from natural borate ores.

ASTM C637-09, “Standard Specification for Aggregates for Radiation-Shielding Concrete,” in conjunction with ASTM C33/C33M-11a, “Standard Specification for Concrete Aggregates,” provides general guidance regarding the requirements and considerations for aggregates used in RSC, including mineralogy; density; grading; deleterious substances; soundness; organic impurities; abrasion resistance; and, depending on the aggregate type, fixed water content and water-soluble material. While these ASTM specifications cover physical and chemical testing requirements for aggregates used in RSC, we are unaware of any current guidance for petrographic examination specific to these specialized aggregates.

ASTM C294-12, “Standard Descriptive Nomenclature for Constituents of Concrete Aggregates,” and ASTM C295/C295M-12, “Standard Guide for Petrographic Examination of Aggregates for Concrete,” apply to petrographic examination of aggregates for general use in concrete. However, these standards do not explicitly mention or describe specialized aggregates such as iron ore aggregates or borate aggregates used for RSC. In this article, we describe petrographic examination methods that can be used to evaluate aggregates for use in RSC.

Specifications and Industry Standards

Aggregates used for RSC are specified in ASTM C637. ASTM C638-09, “Standard Descriptive Nomenclature of Constituents of Aggregates for Radiation-Shielding Concrete,” is a useful supplement to ASTM C637 that provides specific descriptions of some common or important naturally occurring and synthetic constituents of aggregates for RSC. Additional recommendations for measuring, mixing, transporting, and placing high-density concretes that are used for radiation shielding can be found in ACI 221R-96, ACI 304R-00, and ACI 304.3R-96.

Aggregate classes

ASTM C638 provides two classes of aggregates for use in RSC: Class 1 (gamma ray shielding) and Class 2 (neutron shielding). Class 1 includes minerals and rocks with high specific gravities. Class 2 includes minerals and rocks that are particularly effective in absorbing neutrons without producing highly penetrating gamma rays—minerals with substantial boron contents. The most common types of Class 1 and Class 2 aggregates are described in Tables 1 and 2, respectively. Figure 1 shows examples from both classes.

Checking for deleterious materials

The aggregates used in RSC should be relatively clean, free of deleterious materials, and chemically inert. Accurate identification and evaluation of these deleterious materials is often the most critical part of petrographic examination. The common types of potentially deleterious materials in aggregates include (based on ASTM C33, ASTM C294, ASTM C295, and Reference 5):
### Table 1:
Common natural Class 1 aggregates for gamma-ray shielding (based on ASTM C638)

<table>
<thead>
<tr>
<th>Name</th>
<th>Most common sources*</th>
<th>Descriptions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hematite</td>
<td>South America, Africa</td>
<td>Most large hematite ore deposits are sourced from altered banded sedimentary formations and rarely from igneous accumulations. Banded formations may contain iron in carbonates or silicates. The impurities associated with hematite include non-ore bedrock and gangue minerals. Sources vary (between and within deposits) in toughness, compactness, amount of impurities, degrees of weathering, and suitability for use as concrete aggregate.</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>Quebec</td>
<td>Ilmenite ore deposits can comprise coarsely crystalline, massive, tough rocks. Many deposits consist of ilmenite disseminated in rock rather than concentrated as a major rock-forming mineral. Common impurities include constituents of the associated gabbroic or anorthositic rocks. Sources vary (between and within deposits) in composition, hardness, and suitability for use as concrete aggregate.</td>
</tr>
<tr>
<td>Goethite</td>
<td>Utah, Michigan</td>
<td>Goethite occurs in sedimentary conditions or forms as a primary mineral in hydrothermal deposits. The deposits vary from hard, tough, massive rocks to soft, crumbling earths; these alterations frequently occur within fractions of an inch.</td>
</tr>
<tr>
<td>Limonite</td>
<td>Utah, Michigan</td>
<td>Limonite is the generic name for hydrous iron oxides of unknown compositions; frequently goethite and probably mixtures of goethite and hematite. Limonites of high iron content are also called brown iron ores. Frequently, they contain sand, colloidal silica, clays, and other impurities.</td>
</tr>
<tr>
<td>Magnetite</td>
<td>Nevada, Wyoming, Montana</td>
<td>Magnetite ore deposits are associated with metamorphic, igneous, or sedimentary rocks, also in association with hematite and ilmenite. Deposits can form dense, tough, usually coarse-grained rocks. The impurities associated with magnetite may include a wide variety of rock-forming and accessory minerals.</td>
</tr>
<tr>
<td>Barite</td>
<td>Nevada, Tennessee</td>
<td>Barite, also known as baryte, occurs in veins transecting many kinds of rocks, concentrated in sedimentary rocks, and as residual nodules in clays formed by the solution of sedimentary rocks.</td>
</tr>
</tbody>
</table>

*Based on Table 11.1 of ACI 304R. Other sources may be available.

### Table 2:
Common Class 2 aggregates for neutron shielding (based on ASTM C638)

<table>
<thead>
<tr>
<th>Name*</th>
<th>Most common source</th>
<th>Descriptions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colemanite</td>
<td>California</td>
<td>Found in evaporite deposits of alkaline lacustrine environments. Common associated minerals include ulexite and other boron minerals, gypsum, calcite, and celestine.</td>
</tr>
<tr>
<td>Borocalcite</td>
<td>Turkey</td>
<td>The borocalcite refers to Turkish borate ores, which are probably ulexite or colemanite or mixtures of the two (ASTM C638). Ulexite is found in evaporite deposits in arid regions; it is frequently associated with colemanite and other boron minerals, glauberite, trona, mirabilite, calcite, gypsum, and halite.</td>
</tr>
</tbody>
</table>

*Gerstley Borate (U.S. Borax Inc.) has been historically referenced as a common source of Class 2 aggregate; however, the supply has been recently discontinued.
• Rock particles that have the tendency to expand disruptively when exposed to water, such as clay lumps and limestone containing expansive clays;
• Siliceous components of aggregate that are known to be associated with potentially deleterious alkali-silica reactivity (ASR);
• Sulfate-containing rock and minerals, including gypsum and anhydrite, that can cause deleterious sulfate reactions in the concrete;
• Rock particles that have tendency to break into numerous smaller pieces due to inherent weakness of the particles themselves, including clay lumps, poorly indurated shale, and soft sandstone; and
• Organic impurities such as coal, lignite, and wood. If significant coatings are present on the aggregate particles, the coatings should be evaluated for the presence of deleterious materials such as readily soluble salts, organic materials, opal, or gypsum. The bond of the coatings to the particles should also be qualitatively evaluated. Table 3 summarizes potentially deleterious materials commonly associated with aggregates used in RSC. Figure 2 shows thin section photomicrographic examples of deleterious materials.

Identification of metallic phases

The high-density aggregates used in RSC are produced from ore deposits, which contain metallic opaque phases providing the shielding properties to the RSC. The aggregates also contain various amounts of nonmetallic phases, which can include both innocuous non-ore bedrock/gangue minerals and deleterious materials.

As indicated in Section 7.5 of ACI 221R, “In most cases, the exact chemical composition of heavyweight aggregates is not critical as long as the required density is met.” However, if needed, the metallic opaque phases are
identified and quantified by a combination of petrographic and chemical techniques: reflected light microscopy, scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDX), X-ray fluorescence (XRF), and X-ray diffraction (XRD). Impurities or deleterious materials are best identified using transmitted light microscopy.

Assessing physical properties

Ideally, aggregates for RSC should be physically strong and relatively free of soft, friable, altered, or otherwise unsound particles. Aggregate particles exhibiting the following undesirable features should be separated into groups and reported during the petrographic examination (based on ASTM C295):

- Crumbles or readily breaks into several pieces by a light hammer tap;
- Determined by the examiner to be highly porous and absorptive;
- Shows numerous microfractures, microcracks, or cleavage planes; or
- Exhibits abnormal coloration throughout due to chemical or mechanical alteration.

For some high-density aggregates, ideal physical properties may not be met because of high specific gravities. Some aggregates, such as barite and some iron ores, are brittle and tend to break into smaller pieces (Table 4). Some barite aggregate particles may be considered “soft” by an examiner (the Mohs’ hardness of barite is 2.5 to 3.5, which matches that of calcite). These factors do not, however, preclude the use of such materials, provided it can be demonstrated that the manufactured concrete has properties meeting the specification requirements. Also, other properties, such as freezing-and-thawing resistance of the aggregates, may not be significant if the RSC structure is not exposed to such environments.

Shape and surface texture of aggregate particles need to be evaluated, because they may affect workability of the concrete. Coarse metallic aggregates for preplaced aggregate concrete, for example, should be reasonably free of thin, flat, or elongated pieces of metal.

Specific gravity, grading requirements, and physical property requirements such as abrasion resistance are not a part of petrographic examination and will therefore require additional physical testing.

Suggested Procedures for Examinations

Petrographic examination provides an effective means to detect potentially deleterious and undesirable materials in aggregates. Examinations should be performed under strict quality control procedures by a petrographer with knowledge and expertise gained through experience and training.
Sampling

The composition, abrasion resistance, and density of aggregates may vary by locations within a deposit. Test samples should be taken under the supervision of the owner’s representative, and the sampling should meet the requirements of ASTM C637. Aggregates must be sampled, handled, and shipped to the testing laboratory in a manner that will minimize loss of fines, contamination by foreign material, aggregate breakage, and segregation.

Sample classification

The aggregate sample is to be sieved according to ASTM C136-06, “Standard Method for Sieve Analysis of Coarse and Fine Aggregate.” Results of the sieve analysis are later used in calculating the weighted percentages of individual rock/mineral types described by the petrographic examination (ASTM C295). Each sieve fraction of an aggregate sample is examined and its constituents are identified and classified into the following three categories (refer to Fig. 3):

- Category 1: Particles consisting almost entirely of target or primary phase (metallic opaque minerals or boron minerals for borate aggregates);
- Category 2: Particles consisting of a mixture of target phase and nontarget ancillary materials (nontarget hostrock, gangue minerals, and potentially deleterious materials); and,
- Category 3: Particles consisting almost entirely of nontarget ancillary materials.

Fig. 2: Photomicrographs of thin sections, showing potentially deleterious materials in aggregates for use in RSC (Note: Ir indicates Iron oxides, Cm indicates colemanite, and the arrow in each photomicrograph indicates deleterious material): (a) microcrystalline quartz in an iron ore fine aggregate (image taken with transmitted cross-polarized light; length of field from left to right is about 3.8 mm (0.15 in.)); (b) a clayey particle in a colemanite aggregate (image taken with transmitted cross-polarized light; length of field from left to right is about 0.6 mm (0.02 in.)); (c) periclase inclusion in metallurgical slag aggregate (image taken with transmitted plane-polarized light; length of field left to right is about 1 mm (0.04 in.)); and (d) gypsum particle in an iron ore fine aggregate (image taken in transmitted cross-polarized light; length of field left to right is about 1.5 mm (0.06 in.))
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Classification into these three different categories can be made using macro-examination (visual) and stereomicroscopic examination with dilute hydrochloric acid, a hammer, and scratching implements.

Once the aggregate particles are classified into different categories, particles in each category require different methods and techniques for the examination. The Category 1 particles can be classified into a group using descriptors such as “Opaque Metallic Particles” or “Iron-Bearing Metallic Particles.” As mentioned previously, exact identification and quantification of the opaque mineral phases are not critical. If needed, type and relative abundance of different opaque phases can be qualitatively described based on reflected light microscopy, XRD, or a combination of these.

Some Category 1 particles may contain small, localized patches of nontarget ancillary materials (nontarget hostrock and gangue minerals, which may include deleterious materials). These ancillary materials can be best studied using a petrographic (transmitted light) microscope to qualitatively describe their overall distribution, frequency, and composition. The Category 2 particles are evaluated by a petrographic microscope to identify nontarget ancillary materials and to evaluate for the presence of deleterious materials. Aggregate particles in this category may be further segregated into innocuous or deleterious groups upon the presence or absence of deleterious materials in the impurities. Category 3 particles are identified and reported in accordance with ASTM C294 and C295. Each particle in this category is examined and classified into a rock/mineral type. The separated compositional groups should be additionally examined to determine whether a further separation by physical condition is necessary.

For fine aggregate, thin-sections or powder mounts

<table>
<thead>
<tr>
<th>Name</th>
<th>Mohs’ hardness of pure mineral</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hematite</td>
<td>5.5 and 6.5</td>
<td>Physical properties of rocks may vary considerably. Some are relatively soft and brittle and produce dust in the course of being handled. Some hematite rocks tend to be flaky.</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>5.0 to 6.0</td>
<td>Massive ilmenite deposits can form coarsely crystalline, massive, tough rocks but vary from deposit to deposit.</td>
</tr>
<tr>
<td>Goethite and limonite</td>
<td>5.0 to 5.5 (goethite) 4.0 to 5.5 (limonite)</td>
<td>Deposits range from hard tough massive rocks to soft crumbling earths.</td>
</tr>
<tr>
<td>Magnetite</td>
<td>5.5 to 6.5</td>
<td>Deposits can comprise dense, tough, usually coarse-grained rocks. The crushed aggregate particles may be angular and sharp.</td>
</tr>
<tr>
<td>Barite</td>
<td>2.5 to 3.5</td>
<td>The ore contains a large proportion of relatively soft barite particles that may contain open cracks and cleave readily.</td>
</tr>
</tbody>
</table>

*Based on ASTM C638

**Table 4:**
Properties of common naturally occurring high-density aggregates used in RSC

**Fig. 3:** Photomicrographs of thin sections showing different types (categories) of particles in a high-density coarse aggregate sample (Note: Ir indicates iron oxides and Si indicates silicate minerals): (a) Category 1 particle consisting predominantly of iron-bearing metallic minerals (hematite, magnetite, and ilmenite) (image taken with transmitted cross-polarized light; length of field from left to right is about 1.5 mm (0.06 in.)); (b) Category 2 particle consisting of a mixture of iron-bearing metallic and silicate minerals (image taken with transmitted cross-polarized light; length of field from left to right is about 1.5 mm (0.06 in.)); and (c) Category 3 particle consisting predominantly of silicate minerals (image taken with transmitted cross-polarized light; length of field from left to right is about 1.5 mm (0.06 in.)).
should be prepared on glass slides for each sieve fractions smaller than No. 30 (600 μm). A petrographic microscope should be used to identify and classify the constituents, with specific focus on the presence of deleterious and undesirable substances.

**XRF and XRD**

Chemical methods are of great practical importance in the evaluation of aggregates for RSC. Whole sample chemical analysis by XRF provides useful information on overall purity of the metallic ore aggregates. The relative proportions of silica, alumina, lime, sulfur, potash, and soda are important indicators for types and amounts of nontarget ancillary materials present in the aggregate. XRD can be used in conjunction with XRF to assist in the identification and quantification of the minerals present in aggregates, including the major metallic phases and impurities, such as clays. For accuracy in reporting composition, the data are presented as an element or its oxide (for example, Fe, FeO, or Fe₂O₃). Calculations for determining the thickness of the concrete radiation shields require accurate composition data and identification of components in the concrete mixture.

The type and amount of impurities, however, may preclude XRD/XRF as a means for the successful identification of impurities because:

- Small amounts can be diluted in bulk analyses and thus can be missed when the percentage is under the detection limit of the instrument;
- While XRD can help identify phases, it cannot identify microstructure (this is important because, for example, coarse quartz is not prone to ASR but microcrystalline quartz is); and
- Certain minerals are masked by others in a bulk XRD analysis.

Based on those preceding reasons, petrographic examination is an important practice that provides a great alternative or complementary tool to screen for potentially harmful minerals and substances in aggregates for RSC.

**Interpretation and Report**

When the sample contains constituents known to have specific unfavorable effects on concrete, those constituents should be described qualitatively and, to the extent practicable, quantitatively (ASTM C295). However, the report should not contain conclusions or interpretations beyond the findings of the examination.

The petrographic report may include recommendations regarding additional physical and chemical tests that may be required to evaluate the significance of the adverse properties indicated by the petrographic examination (ASTM C295).

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Quality Assurance Requirements

The laboratory performing nuclear-related testing, including petrographic examination of aggregates used in RSC, should conform to Nuclear Quality Assurance-1 (ANSI/ASME NQA-1) requirements, and the testing should meet the applicable requirements of ANSI/ASME NQA-1, Part II, Subpart 2.5. Written record copies of procedures, reports, qualification records, test equipment records, test deviation or exception records, and inspection and examination records should be prepared before the start of the examination. The examiner (petrographer) must have current training records and certificates of authorization or qualification records confirming his or her ability to perform the specified work. The test laboratory is typically subjected to audit and surveillance in accordance with the client’s quality assurance program.

Knowledge and Expertise

Aggregates used in radiation shielding can be petrographically examined to detect the presence of potentially harmful materials. Relative to normal aggregates for general use in concrete, petrographic examination of aggregates for RSC involves more stringent quality assurance requirements. Results of petrographic examination are part of a suite of test methods used to evaluate an aggregate for its suitability, and most deleterious substance limits are based strictly on construction specifications. In certain cases, interpretation of the test results may require engineering judgment based on the consideration and evaluation of the service records of the aggregates, concrete mixture designs, and the intended performance of the specified concrete.

References

2. ACI Committee 304, “Guide for Measuring, Mixing, Transporting, and Placing Concrete (ACI 304R-00) (Reapproved 2009),” American Concrete Institute, Farmington Hills, MI, 2000, 44 pp.
3. ACI Committee 304, “Heavyweight Concrete: Measuring, Mixing, Transporting, and Placing (ACI 304.3R-96) (Reapproved 2004),” American Concrete Institute, Farmington Hills, MI, 2000, 8 pp.

Received and reviewed under Institute publication policies.