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Magnetic titanohematite minerals in uranium-
bearing sandstones

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Magnetic titanohematite minerals
in uranium-bearing sandstones
Abstract

Detrital grains of the rhombohedral ilmenite (FeTiO_3)--hematite (Fe_2O_3) solid solution series (titanohematites) have been identified by thermomagnetic, reflection microscopic, and X-ray diffraction analysis in six uranium-bearing sandstones in the western United States. Many of the titanohematites are ferrimagnetic and have Curie temperatures ranging from about 70°C to 220°C. Magnetic titanohematite is commonly more abundant than magnetite in many samples and, therefore, should be considered as a major source of the permanent magnetization in these units.

Introduction

The iron-titanium (Fe-Ti) oxide minerals are common as detrital grains in uranium-bearing sandstones and have recently received much attention in studies of sedimentary uranium deposits (Shawe, 1976; Adler, 1973; Adams and others, 1974; King and Austin, 1966; Kendall, 1971; Squyres, 1970; Files, 1970; Reynolds, 1975). These studies have shown that the detrital Fe-Ti oxides are sensitive indicators of the geochemical environments associated with the localization of uranium ore (Reynolds, 1975) and are useful in interpreting the genesis of some deposits (Shawe, 1976; and Adams and others, 1974).

Recent application of geophysical techniques to uranium exploration further underscores the importance of the Fe-Ti oxides in uranium-bearing rocks. Because many of the Fe-Ti oxides are magnetic, recognition of these phases and characterization of their magnetic properties are essential to the interpretation of data from magnetic susceptibility and total magnetic field surveys.

The common Fe-Ti oxide minerals are those of two solid solution series: the cubic ulvospinel (FeTiO_4)-magnetite (Fe_3O_4) series (titanomagnetites) and the rhombohedral ilmenite (FeTiO_3)-hematite (Fe_2O_3) series (fig. 1). Members of the latter series closer to the ilmenite end are called hemoilmenites, those closer to the hematite end ilmenoهماتites, and those in between titanohematites (Stacey and Banerjee, 1974). For simplicity, minerals of the rhombohedral series will be referred to as titanohematites in this paper. Compositions of the Fe-Ti oxides of igneous origin vary between those of the end members depending upon the composition of the melt, variations in oxygen fugacity relative to temperatures of crystallization and during subsequent cooling, and cooling rates (Buddington and Lindsley, 1964). It is important to emphasize these compositional controls in the context of geophysical studies because the magnetic properties of the Fe-Ti oxides are related directly to their compositions.

Despite possessing a potentially wide range in composition, the Fe-Ti oxides commonly have been identified simply as "magnetite" or more simply as "black opaques" in the literature of the geology and geochemistry of uranium-bearing sandstones (King and Austin, 1966; Adler, 1973; Harshman, 1972). "Ilmenite" is discussed along with magnetite by Bowers and Shawe (1961), and Adams and others (1974), but in both studies the two phases were not distinguished petrographically. Even in studies of magnetic surveys of uranium deposits, the data are interpreted and modeled in terms of "magnetite" content of the host rock (Ellis and others, 1968; Dodd and Eschliman, 1971; Scott and Daniels, 1976; Smith and others, 1976).

The purpose of this paper is to report the presence of magnetic grains of the ilmenite-hematite solid solution series that have been found to be abundant constituents in some uranium-bearing sediments, and, as such, are important contributors to the permanent magnetization of these rocks.

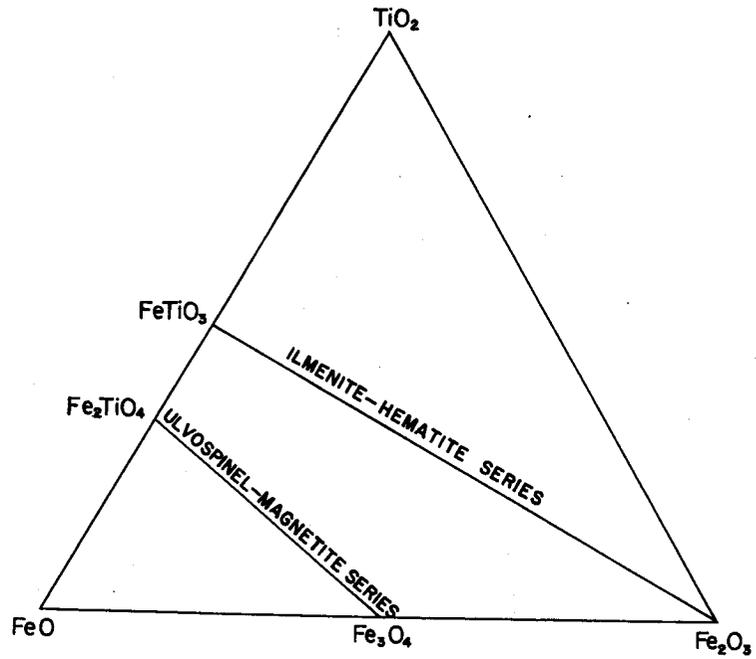


Figure 1.--Ternary diagram showing the subsystem TiO_2 - FeO - Fe_2O_3 and the solid solution joins for the ilmenite-hematite and ulvospinel-magnetite series.

Areas of study

The following discussion is based on studies of five mineralized formations from three of the major uranium-producing districts in the Western United States and from one formation in a district under consideration for development (fig. 2). In order of increasing age these are: (1) the Oakville Sandstone (Miocene) of Live Oak County, southeast Texas; (2) the Catahoula Tuff (Miocene) of Webb County, southeast Texas; (3) the Whitsett Formation (Eocene) of Karnes County, southeast Texas; (4) the Wasatch Formation (Eocene), in the Powder River Basin, Wyoming; (5) the Laramie Formation (Cretaceous), in the Denver Basin, northeast Colorado; and (6) the Morrison Formation (Jurassic) in the San Juan Basin, west-central New Mexico.

Each of the uranium deposits studied represents a simple roll-type orebody except that of the Morrison in which tabular ore has been redistributed into roll forms. Samples were obtained from the outcrop at approximately the same stratigraphic horizon that contained ore (Morrison, Whitsett, and Oakville), from underground (Morrison) and open pit (Whitsett and Oakville) mine walls, and from core samples at depths of about 40-100 m (Wasatch, Catahoula, and Laramie). Although the number of samples from each formation varied greatly, samples from oxidized, reduced, and mineralized rock are represented in each sample suite.

Because magnetic titanohematite grains have been identified in each of the formations, it is important to note that the sediments of the different districts were derived from widely separated source regions. The sediments of the Morrison, Laramie, and Wasatch were primarily derived from Precambrian igneous and metamorphic terrane and older sedimentary rocks, adjacent to their respective basins of deposition (Craig and others, 1955; Goldstein, 1950; and Davis, 1969). Sediments of the three formations of the South Texas Coastal Plain

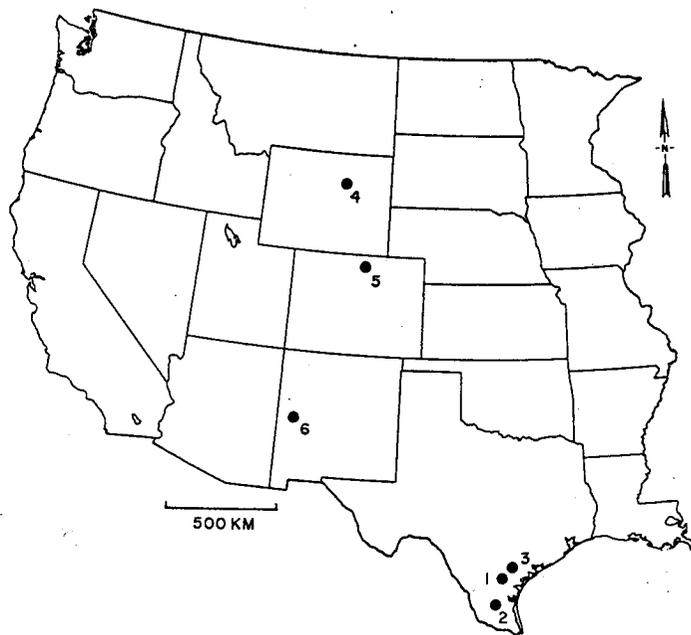


Figure 2.--Map showing location of 6 mineralized formations in western United States. Numbers correspond to formations discussed in text: (1) Oakville Sandstone; (2) Catahoula Tuff; (3) Whitsett Formation; (4) Wasatch Formation; (5) Laramie Formation; (6) Morrison Formation.

apparently have had more diverse origins being comprised in part of detritus from the igneous and sedimentary uplands of Central Texas as well as from intermittent but locally voluminous Early to Middle Tertiary volcanic activity in West Texas and northern Mexico.

Procedures

Magnetic titanohematite was identified primarily by means of thermomagnetic analysis and confirmed by reflected light microscopy of polished grain mounts and by X-ray diffractometry. Because heavy minerals in the sandstones represent only a small portion of the whole sample (about 0.1-5.0 weight percent), it was necessary to obtain the desired fraction by a combination of heavy liquid and magnetic separations. Standard bromoform separation was used to isolate the heavy mineral fraction, and from this, a magnetic separate was obtained either by immersing a strong hand magnet into a suspension of the heavies or by placing the heavies on an inclined plane above a series of alternating electromagnets charged by three-phase induction current.

Thermomagnetic analysis is a method by which magnetization (J) is measured as a function of temperature (T) using a magnetic or Curie balance. Thermomagnetic curves-- J plotted against T --are generated by heating a sample in a furnace placed between the poles of a permanent magnet [2,000 Oersteds (2K0e)], and recording the resulting loss of J until reaching the Curie (or Néel) temperature, the temperature at which a ferrimagnetic (or antiferromagnetic) substance loses its permanent magnetization. Because different magnetic minerals have different Curie temperatures and have characteristic heating and cooling paths on J - T curves, it is possible by thermomagnetic analysis to distinguish among the many magnetic substances. The Curie balance used in this study has

gas-mixing capability (CO_2 and H_2) and a means of continuously monitoring the oxygen fugacity which serves to retard the alteration of the sample during heating--for example, the oxidation of magnetite to hematite (Larson and others, 1975).

The magnetic separate used for study of saturation isothermal remanent magnetization (SIRM) was imbedded in a non-magnetic epoxy chip and placed between the poles of an electromagnet capable of generating fields as great as 10 KOe. Thermal demagnetization to observe the decay of remanent magnetization of the sample after saturation was performed in a non-inductively wound furnace contained in a μ -metal shield in which the ambient field was ± 4 gammas. The specimen was heated and cooled at steps of 100°C, 150°C, and 200°C and the remanence moment measured after each step. The sample could not be heated beyond 200°C which is just below the temperature of decrepitation of the epoxy.

Magnetism in rhombohedral iron-titanium oxides

Magnetism of minerals of the ilmenite-hematite series whose compositions are expressed by $\text{Fe}^{3+}_2 - 2x \text{Fe}^{2+} \text{Ti}_x \text{O}_3$, where the compositional parameter x is the mole fraction of ilmenite, is very complex and incompletely understood (Nagata and Akimoto, 1956; Nagata, 1961; Carmichael, 1961; Westcott-Lewis and Parry, 1971; Hoffman, 1975; Shirane and others, 1959; Ishikawa and Akimoto, 1957; Ishikawa, 1958, 1962). It is clear, though, that Curie temperatures of these rhombohedral phases vary according to composition.

The variation in Curie temperature in relation to composition appears simple and ranges in a linear manner from 680°C for end-member hematite to -218°C for end-member ilmenite, according to studies of Nagata and Akimoto (1956) and Nagata (1961) based on natural and synthetic samples. Westcott-Lewis and Parry (1971), however, analyzed

titanohematites of varying composition synthesized under conditions of controlled oxygen fugacity and found Curie temperatures ranging from 622°C for hematite to -33°C for ilmenite. The 622°C value is well below the widely accepted Néel temperature (680°C) for hematite, and so Westcott-Lewis and Parry (1971) suggested a departure from linearity in the composition range $0.1 > x > 0.0$.

It is difficult to reconcile these differences with the available data and in light of the complexity of the problem. For example, minor element content and previous heating and cooling histories of the natural and synthetic phases apparently can influence Curie temperatures (Ishikawa and Akimoto, 1957; Lindsley, 1976). Recent reviews in rock magnetism, however, implicitly favor the older data of Nagata and Akimoto (Merrill, 1975; Lindsley, 1976). In the following section, compositions of titanohematite derived from thermomagnetic analysis in this study are listed according to the results of both Nagata and Akimoto (1956) and Westcott-Lewis and Parry (1971).

Thermomagnetic analysis of titanohematites from uranium-bearing sandstones

Magnetic titanohematites have been identified by thermomagnetic analysis (a total of 21 separate experiments) in each of the sandstones studied. Thermomagnetic curves, one from each formation, are illustrated on figure 3. Each curve is characterized by a rapid decay of magnetization resulting in the loss of most of the initial moment by 100°C to 200°C. The curves are hyperbolic, concave upward in form and become flat or nearly so (except when magnetite is also present) between 200°C and 300°C indicating low Curie temperatures. Magnetite in minor amounts is identified by small inflexions that flatten by 580°C, the Curie temperature of Fe_3O_4 . Most of the curves are reversible; that is, the cooling path returns

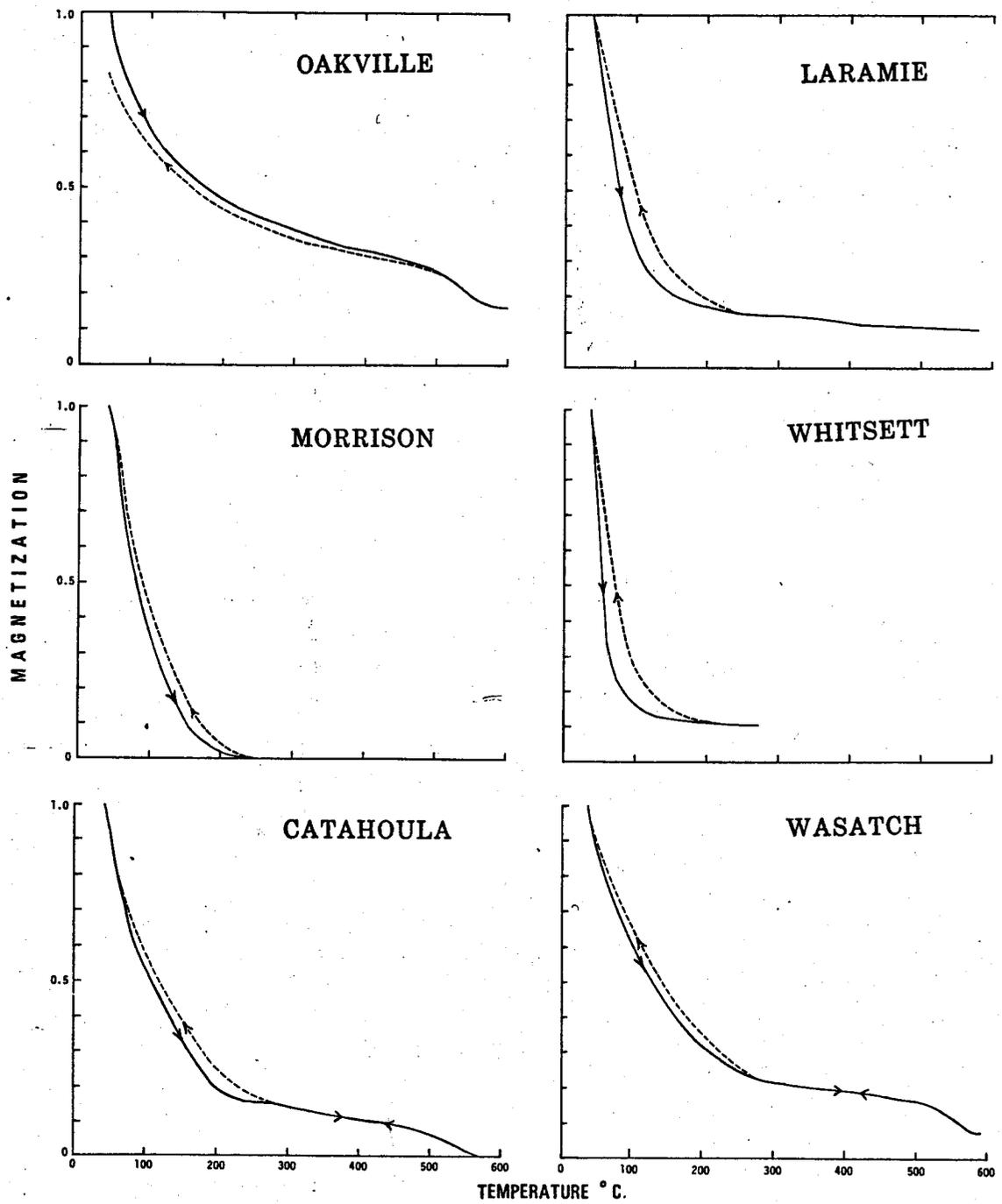


Figure 3.--Thermomagnetic curves of magnetic separates from six mineralized formations. Magnetization (J, normalized to initial moment) plotted against temperature (°C). Solid line represents heating path, dashed line represents cooling path.

to approximately the initial position prior to heating. The offset between the heating and cooling paths is the result of thermal lag in the furnace-sample system. The only exception to this is the J-T curve of the magnetic separate from the Oakville which contains substantially more magnetite than in any of the other samples and which, unlike the other samples, was heated in air to 600°C and maintained at that temperature for 20 minutes. Apparently, oxidation of magnetite to hematite (which has a saturation moment about 0.005 that of magnetite) occurred during heating so that the cooling path thereby returns lower than the heating path.

Thermomagnetic curves of titanohematites are concave upward so that accurate determination of their Curie temperatures is difficult. This difficulty is further compounded if the titanohematites possess a range of Curie temperatures and if magnetite is also present in the sample, as in this study. Two methods are generally used to obtain Curie temperatures of titanohematites: (1) The method of Akimoto (1955) in which the Curie point is determined by extrapolating the initial straight line part of the curve to the temperature axis; and (2) estimating the mid-point of the main inflexion of the curve, the Curie temperature given by the corresponding temperature at this mid-point. Curie temperatures determined by the extrapolation and inflexion methods range from 70°C to 200°C and 100°C to 220°C, respectively. These data along with the corresponding compositional parameters are summarized in table 1.

The strong magnetic response of the titanohematite to weakly magnetized tweezers used to isolate the samples for J-T analysis and to the permanent magnet of the Curie balance strongly suggests that these mineral phases are ferrimagnetic (capable of maintaining a permanent magnetization without the influence of an applied field). This supposition was tested by subjecting a magnetic separate to a strong field in steps of 0.25, 0.50, 0.75, 1.00, 1.50, and 5.00 KOe

Table 1.--Curie temperatures and compositions of titanohematites

[T_c , Curie temperature ($^{\circ}C$); x, composition parameter
 $Fe_{2-2x}^{3+} Fe_x^{2+} Ti_x O_3$.]

Formation	T_c^1	x^3	x^4	T_c^2	x^3	x^4
Whitsett Catahoula Oakville	70-190	0.68-.55	0.84-.66	100-210	0.65-.52	0.80-.63
Wasatch	140-200	.60-.54	.74-.64	210-220	.52-.51	.63-.61
Laramie	120-150	.63-.59	.77-.72	200-220	.54-.51	.64-.61
Morrison	100-150	.65-.59	.82-.72	100-190	.65-.55	.80-.66
ALL	70-200	.68-.54	.84-.64	100-220	.65-.51	.80-.61

¹Extrapolation method (see text).

²Inflexion method (see text).

³After Nagata and Akimoto (1956).

⁴After Westcott-Lewis and Parry (1971).

and measuring the resulting isothermal remanent magnetization (IRM) after each application of the field. If the substance had been paramagnetic (magnetic only in the presence of an applied field) as is end-member ilmenite, the resultant moment after each step would have been nil. Instead, the sample acquired a strong permanent moment at 0.25 KOe and was nearly saturated at 1.5 KOe (fig. 4). Thermal demagnetization of SIRM (acquired at 5 KOe) up to 200°C removed most of the remanence (fig. 4). Demagnetization at 200°C did not eliminate all of the moment partly because the maximum Curie temperature of the sample (determined independently to be 220°C) was not exceeded by heating and possibly because some magnetite may have been present in the tested sample.

Some phases of the ulvospinel-magnetite series can have Curie temperatures within the range of those exhibited by the titanohematites in this study. Curie temperatures range from -150°C for ulvospinel to 580°C for magnetite (Readman and O'Reilly, 1972) so that for titanomagnetites of $0.73 > x \geq 0.55$ ($x \text{Fe}_2\text{TiO}_4 (1-x) \text{Fe}_3\text{O}_4$), Curie temperatures will vary from 70°C to 220°C. Titanomagnetites are readily oxidized, however, to ilmenite, magnetite, and rutile by heating at high temperatures ($>500^\circ\text{C}$) in air. This effect results in irreversible J-T curves, the cooling path returning higher than the heating path. The reversibility of J-T curves of magnetic separates in this study, under both oxidizing and reducing conditions relative to the $\text{Fe}_2\text{O}_3 - \text{Fe}_3\text{O}_4$ buffer when heated to 600°C and immediately cooled, indicates that titanohematites are a major magnetic phase in each separate.

Additional confirmation of titanohematite was made by reflected light microscopic examination of polished sections and by X-ray diffractometry. Polished sections of each separate used for J-T analysis have been examined. These, along with more than 100 other polished sections of magnetic separates of the six mineralized sandstones, contain titanohematite that by both color and anisotropic

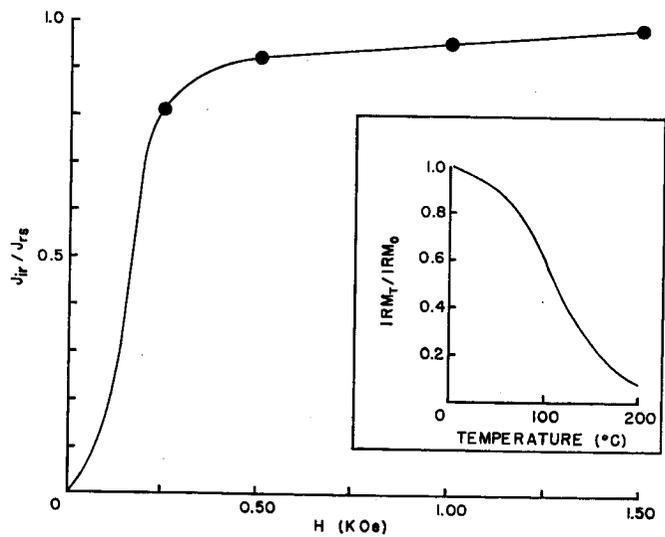


Figure 4.--Diagram showing isothermal remanence (IRM) curve of magnetic separate from Laramie Formation. Ratio of IRM (J_{ir}) to saturation remanence (J_{rs} , acquired at 5.0 KOe) (J_{ir}/J_{rs}) plotted against field H (KOe). Inset diagram shows thermal demagnetization curve of saturation remanence. Ratio of IRM remaining after heating (IRM_T) to initial IRM (IRM_0) (IRM_T/IRM_0) plotted against temperature ($^{\circ}C$).

properties can be easily distinguished from the isotropic minerals of the ulvospinel-magnetite series. Titanomagnetites occur in each of the formations but are for the most part comprised of end-member magnetite divided by lamellae of non-ferrimagnetic ilmenite along (111) crystallographic planes, a texture that results from high-temperature oxidation during initial cooling within the source rock.

Magnetic separates, one each from the Laramie and Wasatch, five from the Whitsett, and seven from the Catahoula were analyzed by X-ray diffractometry. The major diffraction peaks for each separate were intermediate between the principal peaks of ilmenite and hematite. Most peaks in this range were diffuse suggesting that the titanohematites varied in composition. Some diffractograms, however, displayed discrete and well-defined peaks that were closer to the ilmenite d spacings than to those of hematite, a result consistent with the interpretations derived from thermomagnetic analysis (table 1).

Discussion

Identification of the iron-titanium oxide phases is useful in studies of uranium-bearing sediments because the alteration products of these minerals are sensitive indicators of the geochemical conditions under which they form. Moreover, because magnetic properties of rocks can be significantly influenced by titanohematites, recognition of these phases is important for interpreting and modeling the data of magnetic surveys of uranium deposits. The importance of magnetic titanohematites, with regard to geophysical studies, depends upon their abundance relative to the whole rock and to other magnetic phases, particularly magnetite.

Of the uranium deposits considered herein, that in the Catahoula has been most thoroughly investigated. Petrographic

examination of oxidized sandstone at the same stratigraphic horizon as the uranium ore, but which had not been previously sulfidized, indicates that titanohematite and magnetite were deposited in nearly equal abundance. Later alteration, however, of sandstone in proximity to the deposit and under the influence of ore-forming processes, has resulted in greater depletion of magnetite relative to titanohematite. This is primarily because titanohematite is much more resistant to sulfidization than magnetite much of which has been replaced by iron sulfide minerals. Titanohematite also persists under oxidizing conditions that promote relatively rapid oxidation of magnetite to hematite. The net result of these alterations is that titanohematite is the most abundant detrital magnetic phase, and would appear, then, to be responsible for much of the permanent magnetization of these rocks.

Such is apparently the case for the other deposits discussed in this report. For example, titanohematite is the most abundant detrital magnetic phase in the samples of the Morrison Formation. Traces of magnetite have been recorded in some J-T curves, but the magnetite occurs primarily as tiny inclusions within clasts of igneous rocks. Petrographic examination of polished sections and polished thin sections indicates that discrete grains of titanomagnetite and titanohematite may have been initially present in approximately equal amounts, but the iron in the titanomagnetite has been removed leaving merely relict lamellae of non-magnetic TiO_2 (indicative of original ilmenite) along characteristic (111) planes.

Magnetite, along with titanohematite, occurs in the other Tertiary sandstones. Unfortunately, relative abundances of the two phases cannot be accurately determined from J-T curves because the presence of magnetite effectively overshadows the effects of titanohematite when both are present in the same sample. This is

because the saturation moment of magnetite is at least three times greater than that of titanohematite ($x = 0.63$) at room temperature (Westcott-Lewis and Parry, 1971). The greatest deflection in the J-T curves of each magnetic separate corresponds to titanohematite (Curie temperatures $<220^{\circ}\text{C}$) indicating that titanohematite is more abundant than magnetite in these samples.

Although the compositions of titanohematites depend primarily on the initial composition of the parent melt and subsequent cooling history in the source rocks, it is important to consider possible effects of post-depositional low-temperature alteration. For example, studies of ilmenite in beach sands indicate that Fe is selectively removed with respect to Ti (Lynd, 1960; La Roche and others, 1962; Dimanche and Bartholomé, 1976; and Bailey and others, 1956). This process would result in a decrease in the Curie temperature. In this study, titanohematites for J-T analysis were taken from sandstones representing a variety of geochemical environments: reduced rock; rock that at one time was under reducing conditions but was later oxidized during the ore-forming episodes; oxidized rock that had never been reduced; and mineralized rock. No systematic differences in Curie temperature or shape of the J-T curve were observed that could unambiguously be related to geochemical environment. This indicates that if post-depositional changes in the relative abundances of Fe and Ti in titanohematites had occurred, the present method of analysis is insensitive to these changes.

Conclusions

Detrital titanohematite minerals have been identified by thermomagnetic, microscopic, and X-ray diffraction analysis in six uranium-bearing sandstones. Many of the titanohematites are ferrimagnetic and, therefore, are considered to be important in contributing to the permanent magnetization of the sediments in

which they are found. The significance of magnetic titanohematite is further amplified by the observation that magnetite is depleted more readily than titanohematite under the geochemical conditions that control the concentration of uranium in these rocks. The presence of titanohematite, in addition to that of magnetite, should be considered in interpreting data of magnetic surveys of uranium deposits in these sandstones.

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