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2,930,725

NICKEL-IRON ALLOYS

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9 Claims. (Cl. 75—123)

The present invention relates to measuring instruments of the magnetic type and, more particularly, to temperature responsive elements comprised of special nickel-iron alloys which are capable of compensating for errors in the readings of the measuring instruments when the latter are subjected to variations in temperature.

As is well known to those skilled in the art, certain magnetic nickel-iron alloys are particularly useful in making indicating instruments, such as magnetic speedometers, almost completely independent of variations in temperature over an unusually wide range of temperatures. These alloys generally contain about 29% to about 31% nickel and usually much smaller amounts of chromium. They also always contain small amounts of carbon and silicon. The alloys possess a negative temperature coefficient of magnetic permeability that is substantially constant over a range of temperature below the Curie point, this range usually being of the order of 180° F. The martensitic transformation temperature, i.e., the temperature at which there is a phase change from gamma to alpha, may be within or below this range. In practice, the specifications of such alloys require a given Curie temperature and a low martensitic transformation temperature.

However, in the manufacture of these alloys considerable difficulty is encountered in producing alloys of consistent properties, that is to say, the Curie point varies considerably from one heat to another, so that an alloy which should meet the required specification fails to do so. Moreover, the martensitic transformation temperature often varies from heat to heat. When the transformation temperature exceeds that required by a particular specification, the desired high permeability of the alloys at low temperatures is irreversibly reduced when the alloy is inadvertently cooled, during service, below the martensitic transformation temperature, thus impairing the effectiveness of the measuring instrument.

In accordance with the present invention, it has now been found that the irregularities in the properties of the alloys heretofore employed are largely caused by variations in the carbon and silicon contents, particularly in the former. Carbon has commonly been regarded as an element necessarily present and harmless in amounts up to, say, 0.25%. Indeed the carbon content of the alloys normally used is about 0.2%, though it may vary from this by amounts which have been considered negligible but which are in fact important. The silicon content of the alloys has also commonly been about 0.2%.

While having discovered the importance of minor variations in the carbon and silicon contents of the afore-described prior art alloys, to minimize these variations and overcome the disadvantages caused thereby are, in practice, difficult. The carbon content, as will be appreciated by those skilled in the art, is difficult to control within narrow limits, particularly if the alloy is made by melting. If the carbon content is merely reduced, the martensitic transformation temperature is raised, so that the alloy irreversibly loses its high permeability if ex-

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posed to the low temperatures to which it is often liable to be subjected in use. If in turn the proportion of iron is reduced, in order once more to lower the transformation temperature, the proportion of nickel is automatically increased, which causes an increase of the Curie point, with the result that the requirements of many specifications are unsatisfied.

Although attempts were made to overcome the foregoing difficulties and other disadvantages, none, as far as I am aware, was entirely successful when carried into practice commercially on an industrial scale.

It has now been discovered that the aforementioned transformation temperature can be maintained at a desired low level and the Curie point can be controlled in special iron-nickel alloys which contain certain amounts of at least one element selected from the group consisting of copper and molybdenum.

It is an object of the present invention to provide measuring or indicating instruments which are substantially independent of temperature variation.

Another object of the present invention is to provide measuring or indicating instruments which maintain a high degree of accuracy when subjected in service to temperature variation over a wide range of temperature.

The invention also contemplates providing for use in measuring instruments temperature responsive elements comprised of alloys of special composition which are capable of consistently meeting the properties required by specification standards.

It is a further object of the invention to provide special iron-nickel alloys for use as temperature compensating elements, the alloys being of such composition that the martensitic transformation temperature thereof can be maintained at a desired low level and the Curie point thereof can be controlled so that the requirements of specification standards can be consistently satisfied.

It is another object of the invention to provide a process for accomplishing the foregoing objects.

Other objects and advantages will become apparent from the following description.

Generally speaking, the present invention contemplates providing special iron-nickel alloys for use as temperature compensating elements in magnetic measuring or indicating instruments such that the latter operate almost or substantially completely independently of temperature changes over a wide range of temperatures. The alloys of the present invention are substantially free of carbon and silicon and contain about 28.4% to about 32.5% nickel, at least one element selected from the group consisting of copper and molybdenum, the balance of the alloys being essentially iron. Manganese can also be present in the alloys. The copper and/or molybdenum and any manganese must be present in amounts such that the relationship expressed by the following formula is satisfied:

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo} + 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

Not more than up to 6% copper, up to 10% molybdenum and, if employed, up to 6% manganese can be present in the alloys of the invention and the relationship set forth hereinabove must be satisfied. When the alloys contain but one element selected from the group consisting of copper and molybdenum, the respective amounts for each of these elements are 0.6% to 6% copper and 1% to 10% molybdenum. It is particularly important that the relationship established by the above formula be observed to achieve highly satisfactory results. For example, if the value obtained employing the formula exceeds the upper limit represented by the number 12, the alloys tend to become hard and brittle during processing due to the production of second phases. This in turn would

lead to a reduced degree of compensation and variable results between different batches of the same alloy.

It is advantageous in obtaining highly satisfactory results that the alloys contain 29% to 31% nickel and contain copper and/or molybdenum and any manganese in such amounts that the foregoing formula gives a value between about 4 and 9. When this relationship is observed, a Curie temperature of, for example, 180° F. and a martensitic transformation temperature of less than -80° F. and even down to less than -200° F. can be realized. However, in applications requiring relatively high Curie temperatures, e.g., above about 250° F., operating in the lower end of the range of the relationship set forth hereinbefore, e.g., 1.2 to about 2, gives satisfactory results particularly where nickel contents in the upper end of the nickel range are employed. Such alloys are stable down to at least -80° F. Copper and molybdenum and also manganese serve to reduce the transformation temperature, and variations in the proportions of them produce variations in the Curie point which are much smaller than those brought about by carbon and silicon. As a result thereof, the martensitic transformation temperature can be maintained at a desirably low level, e.g., as low as about -200° F. and the Curie point thereof can without appreciable difficulty be controlled. Thus, the alloys of the present invention overcome the inherent disadvantages of prior art alloys which, because of varying carbon and silicon contents from heat to heat, manifested inconsistent properties, whereby their effectiveness as compensating elements was seriously impaired. By reason of having a balanced composition the alloys of the present invention thus possess stability, i.e., do not undergo change of phase or an irreversible change of magnetic properties, at temperatures as low as -80° F. and as high as 400° F. and are therefore eminently suitable for use in indicating or measuring instruments which may experience very low and very high temperatures, such as aircraft instruments and outdoor domestic electrical supply meters.

As indicated hereinbefore, the nickel content of the alloys is from 28.4% to 32.5%. Nickel in excess of 32.5% leads to a more expensive final product because of raw material cost and also gives rise to an alloy possessing a smaller temperature range of compensation. On the other hand, when the nickel content falls below 28.4% the permeability is reduced so that more of the alloy is required to obtain a given degree of compensation. In addition, if the nickel content is reduced much below 28.4%, it becomes impossible to maintain the martensitic transformation temperature below -80° F. and at the same time keep the Curie temperature at a reasonable value, say 180° F.

The alloys are free or substantially free of carbon and silicon. In any event, the carbon and silicon contents should each be not more than 0.03% and preferably less. The use of chromium in the alloys should be avoided and, if present, chromium should not be present in amounts greater than 0.2%.

In obtaining the most consistent results, alloys containing molybdenum and/or copper are made by powder metallurgical methods. If the alloys contain manganese, they should be prepared by melting under an inert atmosphere. Manganese oxidizes so easily that it is difficult to control the amount to be found in the final alloy, and it reduces the corrosion-resistance of the alloy.

It has been further found in accordance with the invention, that the Curie temperature θ_c , and the transformation temperature θ_A at which the austenite transforms on cooling, are given approximately by the equations:

$$\theta_c = A_1 + 70 \text{ Ni} - 48 \text{ Mn} - 22 \text{ Mo} + 40 \text{ Cu}$$

and

$$\theta_A = A_2 - 54 \text{ Ni} - 90 \text{ Mn} - 36 \text{ Mo} - 36 \text{ Cu}$$

(As used herein and in the appended claims, the temperatures θ_c and θ_A are given in degrees Fahrenheit.)

The values of the constants A_1 and A_2 in these equations depend on the amounts of incidental impurities arising from the source of raw materials and method of production. The constants can thus be determined for a particular method of production and the coefficients for nickel, manganese, molybdenum and copper will then enable any alloy to be made to a required specification.

Since the equations for the temperatures contain four variables in the contents of the elements there is clearly a range of alloys which will possess any particular set of values for θ_c and θ_A . Other considerations such as corrosion-resistance, cost and availability of raw materials can thus be used to fix the final composition of the alloy. Using one powder metallurgical method it was found that the constants A_1 and A_2 were -1900 and 1580, respectively. If, therefore, it is desired to manufacture an alloy for which θ_c and θ_A equalled 140° F. and -160° F., respectively, the equations can be rewritten:

$$140 = -1900 + 70 \text{ Ni} - 48 \text{ Mn} - 22 \text{ Mo} + 40 \text{ Cu}$$

and

$$-160 = 1580 - 54 \text{ Ni} - 90 \text{ Mn} - 36 \text{ Mo} - 36 \text{ Cu}$$

Assuming that fairly good corrosion-resistance is required, it is desirable to have a fairly high molybdenum content, say 3%, and no manganese. Substitution in the foregoing equations leads to two simultaneous equations as follows:

$$\begin{aligned} 70 \text{ Ni} + 40 \text{ Cu} &= 2106 \\ 54 \text{ Ni} + 36 \text{ Cu} &= 1632 \end{aligned}$$

the solution for which is 29.3% nickel and 1.4% copper. The final alloy is therefore 29.3% Ni, 3.0% Mo, 1.4% Cu and the balance iron. If, however, it is considered desirable to economize on raw materials, a nickel content of 28.4% would be chosen. The solution for these equations would then be 28.4% nickel, about 2.9% copper, about 2.86% molybdenum, balance iron. It is not desirable to reduce the nickel content below 28.4%, however, as the constants would no longer remain the same, even for a given method of production. In addition, the permeability at a given temperature would tend to be reduced. Heat treatment is required to render the alloy austenitic, but provided the cooling rate is not very slow, e.g., longer than six hours to cool to 200° C., the heat treatment is not critical.

In carrying the method aspect of the invention into practice in producing a suitable iron-nickel temperature compensation alloy having a specified Curie temperature and martensitic transformation temperature, a reference alloy substantially free of carbon and silicon is prepared by the same method and using the same raw materials as to be used in the production of the iron-nickel compensation alloy. The nickel content of the reference alloy is from 28.4% to 32.5%, the balance of the alloy being iron. Up to 6% copper and/or up to 10% molybdenum and, if used, up to 6% manganese can be present in the alloy. The Curie temperature and martensitic transformation temperature of the alloy are then determined in the usual manner. Having determined the Curie and martensitic transformation temperatures, the constants A_1 and A_2 in the equations for θ_c and θ_A above are determined, the values for Ni, Mn, Mo, and Cu in the equations being the respective amounts of these elements in the reference alloy. The determined constants A_1 and A_2 together with the specified Curie temperature, θ_c , and martensitic transformation temperature, θ_A , are then substituted in the equations referred to hereinabove. An iron-nickel temperature compensation alloy is then prepared having a nickel content, a copper and/or molybdenum content and, if present, a manganese content which satisfy each of the equations having

the specified Curie and martensitic transformation temperatures and determined constants A_1 and A_2 . Of course, the relationship expressed by the formula

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo} + 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

must also be satisfied.

A suitable powder metallurgical method of preparing alloys in accordance with the invention is described in connection with an alloy containing molybdenum and copper in addition to iron and nickel. Pure raw materials of the constituent elements were used as follows: carbonyl iron powder, grade MCP, having a mean particle size of about 5 microns; carbonyl nickel powder, grade A, having a mean particle size of about 5 microns; electrolytic copper powder, -300 mesh, having a mean particle size of less than 50 microns; and hydrogen-reduced molybdenum powder having a mean particle size of about 5 microns. Suitable weights of each of these powders were mixed in a cone-mixer for about 1 hour and this intimate mixture of powders was then compacted into a billet of approximate dimensions $10'' \times 2\frac{1}{2}'' \times 2''$ at a pressure of 50 tons/sq. inch. The billet was then sintered in hydrogen for 4 hours at 600°C . to remove residual oxygen and carbon and finally for 6 hours at 1250°C . to produce homogenization of the alloy. The final alloy billet was then worked by conventional forging and rolling operations with frequent high temperature anneals to strip of suitable thickness for the manufacture of shunts for electrical instruments.

By using the foregoing powder metallurgy technique, in which the alloy does not become molten, the need for deoxidants such as magnesium, titanium, etc., is avoided and there is no pick up of impurities from furnace linings, evaporation of constituent elements, etc. Thus, the final product can be much more closely controlled as regards chemical composition, simply by careful weighing out of the constituent powders at the beginning of the process. While it is preferred to use powder metallurgy methods in preparing the alloys of the present invention, vacuum melting techniques may be suitably employed particularly where facilities for production by powder metallurgical techniques are unavailable.

As will be readily understood by those skilled in the art, the expression "balance" used in referring to the iron content of the alloys of the invention does not exclude the presence of other elements commonly present in such alloys as incidental elements, e.g., deoxidizing and cleansing elements, and impurities ordinarily associated therewith in small amounts which do not adversely affect the basic characteristics of the alloys. If, however, close control of properties is required, a method of production would have to be used, such as powder metallurgy or vacuum melting methods, whereby such impurity levels could be maintained at a low level and furthermore so that composition of the alloy can be accurately predicted from the weights of the initial raw materials.

It is to be observed that the present invention provides temperature compensation elements for use in making indicating or measuring instruments substantially independent of variation in temperature over a wide range of temperature. Not only do the compensation elements of the present invention afford the attainment of accurate results, but accurate results are obtained consistently. No detrimental deviation from a predetermined and required Curie temperature is incurred from one heat to another of the alloys of the present invention. This latter characteristic overcomes a serious drawback of prior art alloys. Test-checking of the alloys to determine the properties thereof is not required in accordance with the present invention. Moreover, desired low martensitic transformation temperatures can be easily achieved in accordance with the invention.

The temperature compensation elements comprised of

the alloys of the present invention are highly suitable for use in magnetic speedometers, watt-hour meters, voltage and current regulators, and other electrical supply meters.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention; as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

I claim:

1. A temperature responsive element for use in magnetic indicating instruments, subjected in service to changes in temperature over a wide range of temperature comprised of a temperature compensating alloy characterized by the property of having a specified Curie temperature and martensitic transformation temperature, said compensating alloy being substantially free of carbon and silicon and containing 28.4% to 32.5% nickel, at least one metal selected from the group consisting of copper and molybdenum in amounts of up to 6% copper and up to 10% molybdenum, up to 6% manganese and the balance being substantially iron, the nickel and metal from the group consisting of copper and molybdenum and the manganese being present in such amounts as to satisfy the relationship expressed by the formula

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo} + 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

and to satisfy each of the following equations:

$$\theta_c = A_1 + 70 \text{ Ni} - 48 \text{ Mn} - 22 \text{ Mo} + 40 \text{ Cu}$$

$$\theta_A = A_2 - 54 \text{ Ni} - 90 \text{ Mn} - 36 \text{ Mo} - 36 \text{ Cu}$$

wherein A_1 and A_2 are constants, and θ_c and θ_A are the specified Curie temperature and martensitic transformation temperature, respectively.

2. A temperature compensating alloy for use in magnetic measuring instruments subjected in service to temperature changes comprising up to but not more than 0.03% carbon, up to but not more than 0.03% silicon, 28.4% to 32.5% nickel, at least one metal selected from the group consisting of copper and molybdenum in amounts of up to 6% copper and up to 10% molybdenum, up to 6% manganese, the balance of the alloy being substantially iron, the amounts of copper, molybdenum and manganese satisfying the relationship expressed by the formula

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo} + 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

said alloy being further characterized by being capable of meeting a specified Curie temperature and martensitic transformation temperature, when the same amounts of nickel, copper, molybdenum and manganese are so correlated that each of the following equations are satisfied:

$$\theta_c = A_1 + 70 \text{ Ni} - 48 \text{ Mn} - 22 \text{ Mo} + 40 \text{ Cu}$$

$$\theta_A = A_2 - 54 \text{ Ni} - 90 \text{ Mn} - 36 \text{ Mo} - 36 \text{ Cu}$$

wherein A_1 and A_2 are constants and θ_c and θ_A are the specified Curie temperature and martensitic transformation temperature, respectively.

3. A temperature responsive element capable of compensating for errors in the readings of magnetic measuring instruments when subjected to temperature variation during service, said temperature responsive element comprising a compensating alloy containing 29% to 31% nickel, not more than 0.03% carbon, not more than 0.03% silicon, at least one metal selected from the group consisting of copper and molybdenum in amounts of from 0.6% to 6% copper and from 1% to 10% molybdenum, from 0.6% to 6% manganese, the amounts of copper,

molybdenum and manganese satisfying the relationship expressed by the formula

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo}$$

$$+ 2 \times \text{percent Mn} = 4 \text{ to } 9$$

and the balance of the alloy being substantially iron.

4. A temperature responsive element capable of compensating for errors in the readings of magnetic measuring instruments when subjected to temperature variation during service, said temperature responsive element being comprised of an alloy substantially free of carbon and silicon and containing 28.4% to 32.5% nickel, at least one metal selected from the group consisting of copper and molybdenum in amounts of up to 6% copper and up to 10% molybdenum, up to 6% manganese, the amounts of copper, molybdenum and manganese satisfying the relationship expressed by the formula

$$2 \times \text{percent Cu} + 1.2 \times \text{percent Mo}$$

$$+ 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

and the balance of the alloy being substantially iron.

5. A temperature responsive element as described in claim 4 wherein the metal selected from the group consisting of copper and molybdenum is copper.

6. A temperature responsive element as described in claim 4 wherein the metal selected from the group consisting of copper and molybdenum is molybdenum.

7. A temperature responsive element as described in claim 4 wherein both copper and molybdenum are present in the alloy.

8. A method for producing a magnetic iron-nickel temperature compensation alloy having a specified and pre-required Curie temperature and martensitic transformation temperature comprising the steps of establishing a reference alloy composition substantially free of carbon and silicon and containing 28.4% to 32.5% nickel, up to 6% manganese, up to 10% molybdenum, up to 6% copper, and the balance essentially iron, the amounts of nickel, manganese, molybdenum and copper in said reference alloy composition being within their respective ranges such that the relationships expressed by the following equations:

$$(1) \quad A_1 = \theta_c - 70 \text{ Ni} + 48 \text{ Mn} + 22 \text{ Mo} - 40 \text{ Cu}$$

$$(2) \quad A_2 = \theta_A + 54 \text{ Ni} + 90 \text{ Mn} + 36 \text{ Mo} + 36 \text{ Cu}$$

are satisfied, θ_c and θ_A being the Curie temperature and martensitic transformation temperature, respectively, of said reference alloy composition and A_1 and A_2 being constants; and thereafter producing the iron-nickel temperature compensation alloy by the same method and with the same raw materials as employed in producing the reference alloy, said temperature compensation alloy being characterized in that it is substantially free of carbon and silicon and contains 28.4% to 32.5% nickel, and at least one metal selected from the group consisting of molybdenum and copper in the ranges of up to 10% molybdenum and up to 6% copper, and up to 6% manganese, and being further characterized in that the amounts of nickel, molybdenum, copper and manganese are present within their respective ranges such that the relationships expressed by the following equations:

$$(3) \quad A_1 = \theta_c - 70 \text{ Ni} + 48 \text{ Mn} + 22 \text{ Mo} - 40 \text{ Cu}$$

$$(4) \quad A_2 = \theta_A + 54 \text{ Ni} + 90 \text{ Mn} + 36 \text{ Mo} + 36 \text{ Cu}$$

$$(5) \quad 2 \times \text{percent Cu} + 1.2 \times \text{percent Mo}$$

$$+ 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

are satisfied, θ_c and θ_A being the desired Curie temperature and martensitic transformation temperature, respectively, and A_1 and A_2 being the constants of Equations 1 and 2 above, whereby there is provided a magnetic iron-nickel temperature compensation alloy having a specified and pre-required Curie temperature and martensitic transformation temperature.

9. A method for producing a magnetic iron-nickel temperature compensation alloy having a specified and pre-required Curie temperature and martensitic transformation temperature comprising the steps of establishing a reference alloy composition containing up to about 0.03% carbon, up to about 0.03% silicon, 28.4% to 32.5% nickel, up to 6% manganese, up to 10% molybdenum, up to 6% copper, and the balance essentially iron, the amounts of nickel, manganese, molybdenum and copper in said reference alloy composition being within their respective ranges such that the relationships expressed by the following equations:

$$(1) \quad A_1 = \theta_c - 70 \text{ Ni} + 48 \text{ Mn} + 22 \text{ Mo} - 40 \text{ Cu}$$

$$(2) \quad A_2 = \theta_A + 54 \text{ Ni} + 90 \text{ Mn} + 36 \text{ Mo} + 36 \text{ Cu}$$

are satisfied, θ_c and θ_A being the Curie temperature and martensitic transformation temperature, respectively, of said reference alloy composition and A_1 and A_2 being constants; and thereafter producing the iron-nickel temperature compensation alloy by the same method and with the same raw materials as employed in producing the reference alloy, said temperature compensation alloy being characterized in that it contains not more than 0.03% carbon, not more than 0.03% silicon, 28.4% to 32.5% nickel, and at least one metal selected from the group consisting of molybdenum and copper in the ranges of 1% to 10% molybdenum and 0.6% to 6% copper, and up to 6% manganese, and being further characterized in that the amounts of nickel, molybdenum, copper and manganese are present with their respective ranges such that the relationships expressed by the following equations:

$$(3) \quad A_1 = \theta_c - 70 \text{ Ni} + 48 \text{ Mn} + 22 \text{ Mo} - 40 \text{ Cu}$$

$$(4) \quad A_2 = \theta_A + 54 \text{ Ni} + 90 \text{ Mn} + 36 \text{ Mo} + 36 \text{ Cu}$$

$$(5) \quad 2 \times \text{percent Cu} + 1.2 \times \text{percent Mo}$$

$$+ 2 \times \text{percent Mn} = 1.2 \text{ to } 12$$

are satisfied, θ_c and θ_A being the desired Curie temperature and martensitic transformation temperature, respectively, and A_1 and A_2 being the constants of Equations 1 and 2 above, whereby there is provided a magnetic iron-nickel temperature compensation alloy having a specified and pre-required Curie temperature and martensitic transformation temperature.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,930,725

March 29, 1960

Charles Alfred Clark

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 57, in the formula after "12" insert a period; column 5, lines 54 and 55, for "impulity" read -- impurity --; line 56, after "that" insert -- the --; column 6, line 52, in the formula after "12" insert a comma; column 7, line 4, in the formula after "9" insert a comma; line 19, in the formula after "12" insert a comma; line 51, for "wtih" read -- with --; column 8, line 20, for "manganeses" read -- manganese --; line 41, for "with" read -- within --.

Signed and sealed this 11th day of April 1961.

(SEAL)

Attest:

ERNEST W. SWIDER
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Acting Commissioner of Patents